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STABILITY OF THERMAL CONTROL COATINGS EXPOSED TO COMBINED SPACE ENVIRONMENTS

By William R. Holland

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AVCO ELECTRONICS DIVISION
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for

AMES RESEARCH CENTER
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ABSTRACT

An in situ reflectometer system was designed, fabricated and installed in an existing Avco space simulation facility. After installation of the in situ system, qualification tests were conducted on the system. Tests were conducted on twelve materials including combined environment of vacuum, protons and ultraviolet, protons and vacuum only, ultraviolet and vacuum only and vacuum only. The ZnO/K2SiO3 was found to be more susceptible to protons and less to ultraviolet radiation and the La2O3/K2SiO3 more susceptible to ultraviolet radiation with very little damage by protons. Of the powdered samples tested only the TiO2 showed measurable damage with protons only irradiation.

FOREWORD

This draft report was prepared by the Avco Electronics Division, Tulsa Operation, as the final documentation of work performed under Contract NAS2-3646, issued by National Aeronautics and Space Administration, Ames Research Center, Moffett Field, California. Mr. Elmer Streed was Technical Monitor.

This report covers work performed from June 1966 to August 1967.

Prepared By:

W. R. Holland, Chief

Environmental Physics Section

Approved By:

D. G. Crews

Manager of Engineering

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I. <u>INTRODUCTION</u>

Reliable spacecraft thermal design requires a knowledge of solar absorptance of all spacecraft surfaces exposed to outer space and the stability of the thermal properties of these surfaces in the total space environment. Furthermore, this information must be obtained under conditions as near to outer space as possible, i.e., simulation and measurement without return to the atmospheric conditions.

This is borne out by the fact that during the course of work of many investigators (1-7) effects were noticed which were modifying data upon exposure of the samples to atmospheric environments.

A rapid bleaching of damage was taking place in some materials when exposed to atmosphere. This bleaching was masking the true results of the test and was leading to erroneous conclusions. In order to properly investigate the effects of space environment, certain improvements had to be made in the equipment. It became apparent that an in situ capability for making reflectance measurements was necessary.

The primary aim of NASA Contract NAS2-3646 was to fabricate equipment and perform the investigation of space degradation on selected thermal control coatings in situ. The parameters which could be simultaneously simulated were solar electromagnetic radiation, charged particles, ultrahigh vacuum and equilibrium temperature. Of these, the parameters chosen as variables were the electromagnetic radiation, proton energy and proton flux.

The first half of the contract was spent in the design and fabrication of in situ equipment and the last half in the performance of the tests to qualify the equipment and test coating materials as prescribed by NASA.

The system as fabricated under this contract was adapted to an existing chamber at Avco/Tulsa and is described under Section III, Test Equipment. It provides for multi-sample irradiation and selective transfer to the integrating sphere for reflectance measurements. In addition, during the time of fabrication of the in situ equipment, tests were conducted of non-in situ type on materials which were considered more stable. The results of these tests are reported in the pages following the results of the in situ tests.

II. PROGRAM SCOPE

The program consisted of two phases: (1) Design, fabrication and qualification of test equipment, and (2) in situ testing of designated materials. These phases are described in the following paragraphs.

A. Phase I - Design, Fabrication and Qualification

The scope of this portion of the program was the design of a mechanism which would adapt to an existing vacuum chamber and transfer four 15/16-inch diameter selective samples from the irradiation position to the reflective measurement position. The sample was to operate over a temperature range of -100°F to 300°F position and the samples easily removable for transfer. In addition, various accessory items were required in order to adapt the existing beam line and monochromator to the in situ chamber system.

The integrating sphere was to be mounted on the end of the vacuum chamber coated inside with magnesium oxide and having vacuum sealed windows for light input and detectors. This position was selected for the sphere because it was felt that (1) it would be more difficult to feed the light from the monochromator and optical switch into the sphere, (2) the detectors require special consideration to prevent outgassing problems, and (3) no vacuum tight electrical feedthroughs through the chamber were necessary if the sphere is on the outside.

The qualification was to consist of comparison measurements against a Gier-Dunkle integrating sphere over a wavelength range of 0.30 to 2.5 microns for both diffuse and specular samples. The diffuse samples were specified as $\rm ZnO/K_2SiO_3$ and the specular samples as $\rm SiO$ on vapor deposited aluminum. Five sets of measurements were required on each sample and the mean spectral deviation determined to demonstrate the reproducibility of the equipment.

B. Phase II - Test Program

1. Sample Materials

The materials which were selected for in situ test were ZnO/K₂SiO₃, (two batches F-1-47-D and 1-11-9-13), La₂O₃/K₂SiO₃ (two batches F-1-53A and F-1-38), S-13G, ZnO/Silicone, TiO₂/Silicone and powdered samples of MgO, TiO₂, SiO₂, Al₂O₃ and La₂O₃. Non-in situ radiations were conducted on SP500 ZnO powder and vacuum deposited silver on fused silica. Test specimens are described in Table I.

2. Description of Irradiation Tests

In the majority of the in situ irradiation tests, one sample was to be subjected to an environment including vacuum and near ultraviolet. A second sample was to be subjected to a combined environment including proton irradiation, ultraviolet and vacuum ultraviolet. A third sample was to be subjected to protons radiation only and the fourth sample was to be used as a control sample and incurred only vacuum. The following parameters were to be used in the tests:

a. Near Ultraviolet Radiation

Ultraviolet radiation on the 2000-4000 angstrom range provided by a 5 KW xenon lamp installed in the solar simulator for the first three tests and a 5 KW mercury xenon lamp for all non—in situ tests and the remainder of the in situ tests. It was determined during the program that the xenon lamp would provide various irradiance values from 5.5 solar constants to 7.5 solar constants. The Hg Xe lamp was to operate at a rate of 10 solar constants. Total ultraviolet exposure was to be 750 sun hours with both lamps.

b. Vacuum Ultraviolet Radiation

Radiation in the 1050 to 2000 angstroms range was to be supplied by a hydrogen discharge lamp at 10 solar equivalents of Lyman-alpha for 75 hours per test.

SPECIMEN	SOURCE	PURITY %	PAR TICLE SIZE	COMMENTS
AL ₂ O ₃	Linde Division Union Carbide Cómpany	86.66	o. 05µ	Metallurgical Grade Pressed into recessed AL holder 1/16" deep, with black coating, at 2000 psi, covered with quartz disk until exposed
Lå203	Rare Earth Division American Potash and Chemical Corporation	66.66	3.5µ	Code 529, hygroscopic, could be partially La(OH) ₃ at surface
MgO	Vitro Labs	99.41	0. 02 5 և	Sub micron size, Plasma arc product
$\sin c_2$	Thermal American Fused Quartz Co.	~ 2.66	~ 40,և	"Spectrosil" Grade or Better
TiO_2	E. I. DuPont DeNemours and Company (Inc.)	88 TiO ₂ 0. 3% Max. AL ₂ O ₃ 8% Max. SiO ₂	0.22n O ₃	Ti Pure R-960 slightly hygroscopic This TiO ₂ has a SiO ₂ coating which increases particle size $< 0.1 \mu$
Z_{nO}	New Jersey Zinc Co.	66.66	0.25-0.35րև	SP-500, Super purity
S-13G	This was a typical S-13G sample composition supplied by Jet Propulsion Laboratory.	3G sample by Jet Propulsion		

TABLE I - TEST SPECIMENS DESCRIPTION

SPECIMEN	SOURCE	COMMENTS
1-11-9-13	S.P 500 ZNO New Jersey Zinc Co. P.S 7 (K ₂ SiO ₃) Sylvania Electronics (35% Solids)	P.B.R. = 2.15 Ball Milled 2 Hours Sprayed ~ 6 coats oven cured 150°C for 1 hour. Thickness ~ 6 mil. Paasche type AUTF Air Brush used to apply all coatings.
F-1-47D Binder P.S7	S.P 500 ZNO P.S 7 (K_2SiO_3) Same as above	P. B.R. = 5.2 Ball Milled 4 hours Sprayed 6 coats ~ 4 mil - Oven cured 150°C ZNO calcined 1200°F 16 hours cured vac. oven 29" HG & 150°C 1 hour
F-1-55	S. P. 500 ZNO R. T. V 602 General Electric Silicone N-Butanol (Reagent Gr) Mallinckrodt Chemical	P.B.R. = 2.4 Ball Milled 2 hours Sprayed 6 coats - Oven cured 150°C 1 hour 6.5 Mil thickness - P.V.C. = 30% Undercoat SS4044 (General Electric) Silicone Primer - Sprayed ~ 5 mil
F-1-38 Binder P.S7	LA ₂ O ₃ Code 529 -7 Rare Earth Division American Potach & Chemical Corporation	P.B.R. = 5 LA_2O_3 Calcined 2000^{0}F - 16 hours Ball Milled 4 hours - Sprayed 6 coats Cured at Ambient $\sim 6 \text{ mil}$ LA ₂ O ₃ converted to LA(OH) ₃ during ball milling
F-1-53A Binder P.S 7	$\mathrm{LA_2O_3}$ Code 529	P.B.R. = 1.75 LA ₂ O ₃ Silicate treated (Encapsulated in $K_2^SiO_3^S$) - Oven dry 150°C 1 hour Ball Milled 2 hours - Sprayed 8 coats ~ 4 mil thickness
F-1-56	TiO ₂ R-960 Ti Pure E.I. DuPont DeNemours and Company (Inc.) R.T.V 602 G.E. N-Butanol (R.G.)	PB.R. = 2.5 Ball Milled 3 hours Oven cured 300°F - 1 hour P.V.C. = 34.7% Sprayed 9 coats ~ 8 mil thickness Undercoat SS4044 (General Electric) Silicone Primer - Sprayed ~ 5 mil

TABLE I - TEST SPECIMENS DESCRIPTION

c. Protons

All of the tests were to be conducted with 10 Kev protons. Total proton fluxes were to vary from 2×10^{15} p/cm² to 1×10^{16} p/cm².

In some tests proton irradiations were to be carried out at a continuous low flux rate for 75 hours, and in other tests, "accelerated", a higher flux rate was to be used at the beginning of the test.

d. Sample Temperature

Sample temperatures were to be maintained near 298°K for the various tests.

e. Chamber Pressure

No test was to be initiated until pressure in the test chamber was 10^{-7} torr or less.

3. Determination of α_s and $\Delta \alpha_s$

Spectral reflectance measurements were to be made on the samples before and after each irradiation in the <u>nonin situ</u> tests and before, during, and after irradiation in the <u>in situ</u> testing. Spectral reflectance curves were to be plotted from these data.

Solar absorptance values were to be obtained by averaging representative values of spectral reflectance obtained from the curves for each two percent extraterrestial solar energy increment in the measured wavelength range and substracting these average values from unity. The solar absorptance values presented will not be total values relating to the entire spectrum, since this would require measured spectral reflectance values to 3.9 microns which is beyond the measured range of the detectors used. The values given will be related to the measured wavelength range.

III. TEST EQUIPMENT

Avco's space simulation facility is shown schematically in Figure 1. It consists of an ultra-high vacuum chamber with the newly designed in situ reflectometer system, solar simulator, transition section and Van de Graaff accelerator. The various components of the system, beginning with the new items designed under this contract, are described in the following paragraphs.

A. <u>In Situ Reflectometer System</u>

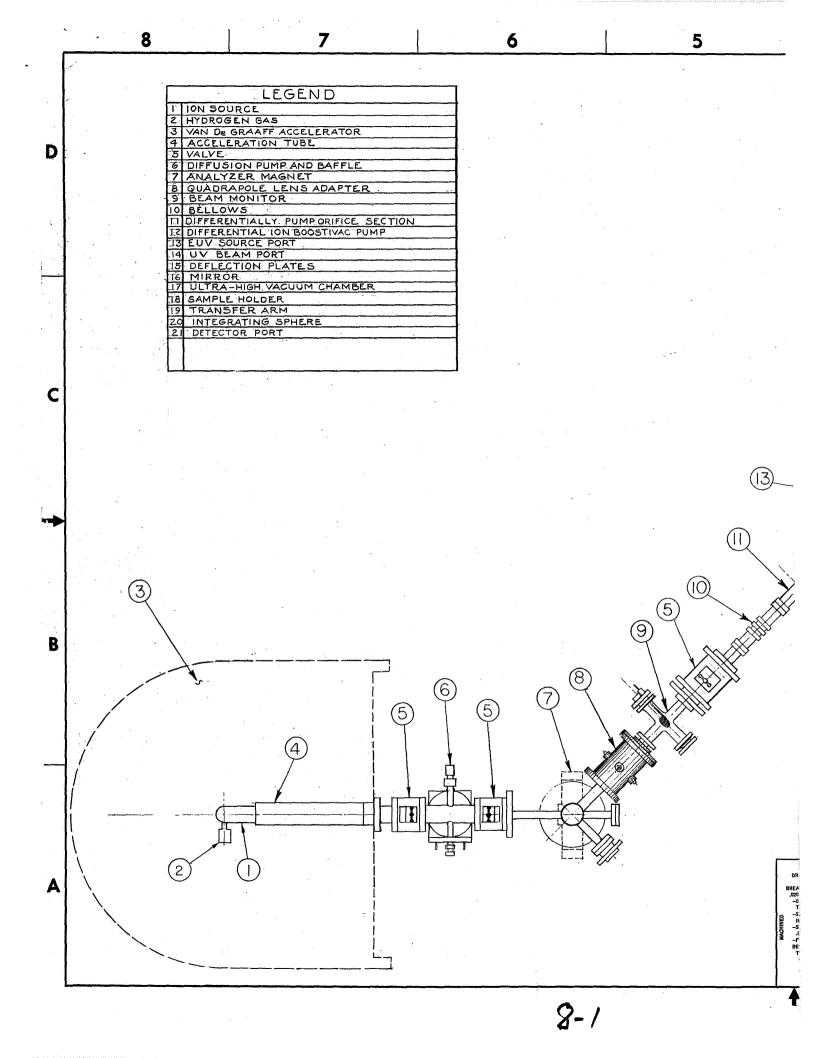
The system was designed to selectively transfer any one of four samples in the sample holder (irradiation position) to the integrating sphere or vice versa. This may be accomplished at pressures of 1×10^{-7} torr or less. It was designed to fit an existing Avco ultra-high vacuum chamber. Dimensional information for the various components is given in the appendix.

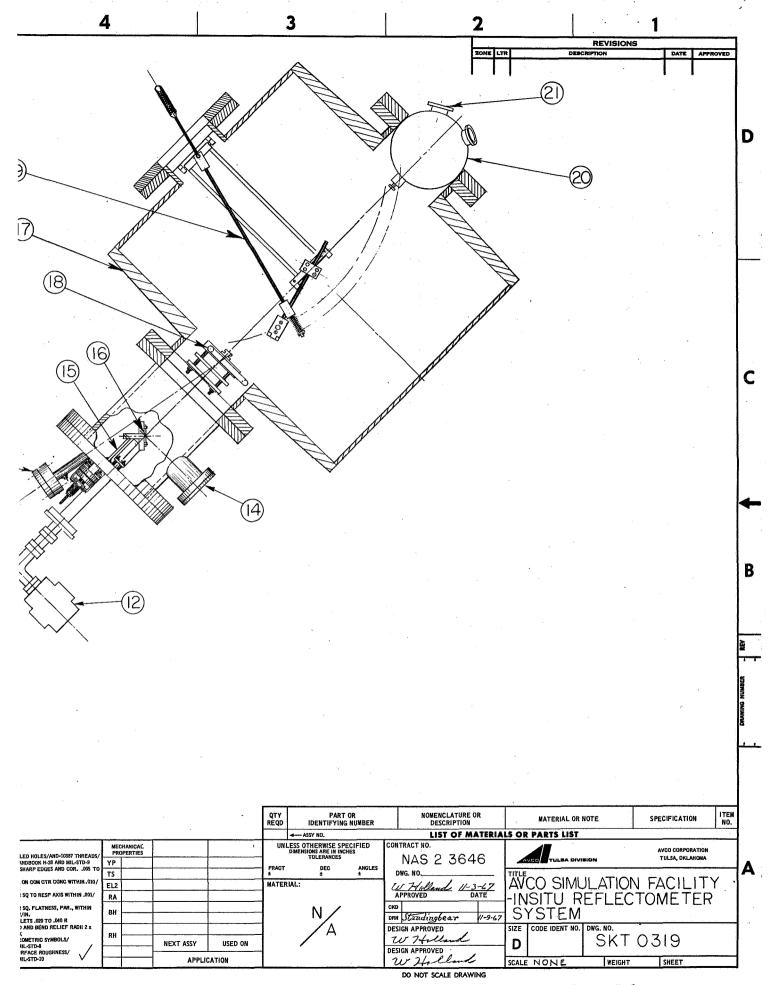
1. Transfer Arm Assembly

The transfer arm, shown in Figure 2, (Item 19, Figure 1), is mounted on a large flange at the side of the chamber and a bellows provides the necessary vacuum seal. The transfer arm is pivoted for movement in two directions and stops are provided to prevent over travel. The arm is coupled to a slider bar to which is attached "U" shaped fingers. These fingers will fit around a protrusion on the conical plugs on which the sample is fastened. The slider bar slides in a pivoted retainer mounted on arms also pivoted at the gimball mount on the flange. When the slider bar is caused to slide back against a spring stop, further movement of the transfer arm causes the arm to swing in an arc and thus turn the sample 180°. This movement of the transfer arm in an arc of approximately 60° will cause the sample to be removed from the sample holder and plugged in to the integrating sphere or vice versa.

2. Sample Holder Assembly

The sample holder assembly which was designed and fabricated for this study is shown in Figure 3 (Item 18, Figure 1). It is a large copper block which has four





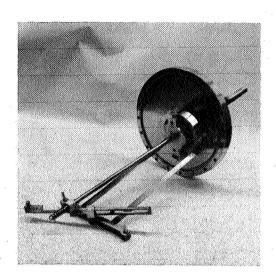


Fig 2 - Transfer Arm

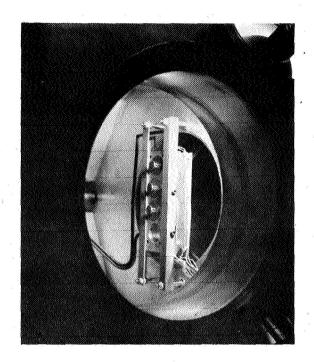


Fig 3 - Sample Rack, Mounted In Chamber

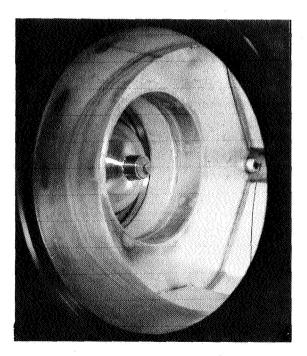


Fig 4 - Integrating Sphere-Vacuum View

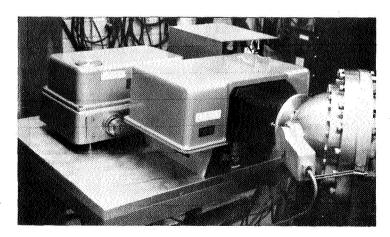


Fig 5 - Integrating Sphere - Atmosphere View

conical shaped holes arranged in a slight vertical arc because of the motion of the transfer arm. The samples can be clamped to conical plugs which fit in the holder. Three samples face the sources of radiation and one, mounted in the bottom of the holder, is shielded from these sources. Coolant fluids can be circulated through the sample holder via stainless steel tubing running through an insulated feedthrough which isolates the holder from ground. A cartridge heater is installed in the holder for maintaining higher temperatures. A thermocouple is spring loaded against the back of the sample which is irradiated by both particulate and electromagnetic radiation.

The samples tested are nominally 15/16-inch in diameter. The sample area which is exposed to radiation is 13/16-inch in diameter, since the samples are clamped at the edge. Two plates are mounted on insulating standoffs from the face of the sample holder. The plate farthest from the holder faces serves to collimate the particle beam and thus define the area of sample and sample holder which is irradiated. Two 15/16-inch diameter holes in this plate are centered over the samples. The charged particle beam as it is incident on this plate is rectangular, approximately 1-1/8-inches long x 3/16-inch wide. The beam is rastered across the appropriate collimating hole in the plates by the sawtooth voltage applied to the deflection plates. The collimating plate is connected to an insulated feedthrough in the four-inch vacuum flange and may be grounded or biased as necessary. The purpose of the intermediate plate is to serve as a secondary electron suppressor. The charged particle beam passes through a short section of tubing in this plate. The plate (and tubing) is biased negatively with respect to the sample holder and collimating plate to force secondary electrons arising from particle bombardment back to their respective sources. Target current is read directly by connecting a meter to the insulated feedthrough which supports the sample holder. When samples which are good insulators, such as the paints, are being irradiated, a grid consisting of five parallel strands of one mil stainless wire is mounted in contact with the sample surface. This grid masks less than one percent of the sample area and serves to prevent the sample surface from becoming electrically charged with consequent "chasing" of the particle beam and inaccurate beam current readings.

3. Integrating Sphere

The integrating sphere is shown in figures 4 and 5 (Item 20, Figure 1). It was fabricated from two stainless steel hemispheres to which were welded mounting flanges to mount on the chamber. It has three ports, one for the sample, one for the detector and one for the light from the Perkin-Elmer Model 112U spectrophotometer. It is coated inside with magnesium oxide and a baffle is mounted inside the sphere to prevent the light from the sample from seeing the detector directly.

B. Vacuum Chamber

The Avco ultra-high vacuum chamber is shown schematically in Figure 6 (Item 17, Figure 1). It has a normal working volume of 24" x 24" in the form of a right circular cylinder. This chamber has a ratio of total volume to useful volume of approximately 90%. The chamber was fabricated and welded with low carbon stainless steel. Ports entering into the vacuum chamber itself are metal gasketed and are 10.75" in diameter for access purposes or 1.5" in diameter for instrumentation. In addition, a four-inch port is provided for electrical feedthroughs. The chamber is pumped with 900 liters/sec of cold cathode ion getter pumping system. The system is comprised of a high voltage source, a magnet assembly, and two active pumping element assemblies. The magnetic field in the region of the pump cells is approximately two kilogauss provided by the magnet assembly. The chamber is a double wall chamber and the annulus between the cylinders, except in regions taken up by feedthroughs and flanges, serves as a passage for liquid nitrogen or other coolants to provide cold surrounds and some cryogenic pumping if desired.

C. Van de Graaff Accelerator

The accelerator (Item 3, Figure 1) is capable of accelerating positive ions or electrons in the energy range from about

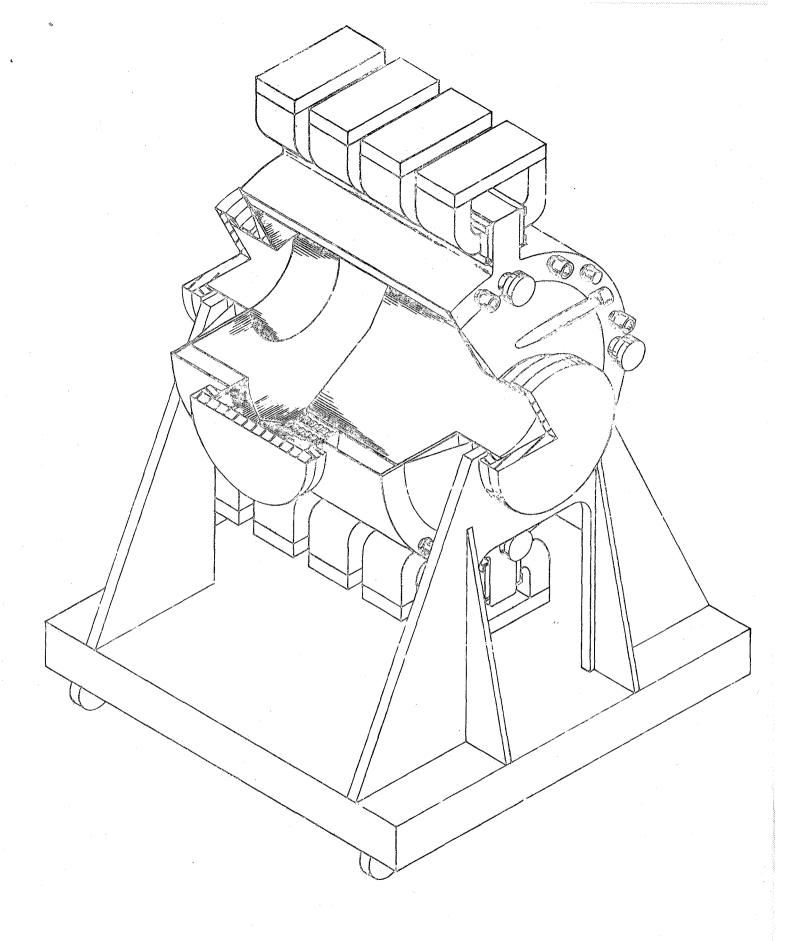


Figure 6 - WALL-PUMP ULTRA-HIGH VACUUM SYSTEM

10 Kev to 500 Kev. The Van de Graaff voltage generator is used from about 500 Kev down to 100 Kev, below which point the voltage stability becomes poor. Energies below 100 Kev are provided by disabling the Van de Graaff voltage generator in the accelerator and connecting an auxiliary 0 - 100 kilovolts power supply across the acceleration tube. Positive ions are generated by an R.F. ion source. The accelerator is pumped by a four-inch oil diffusion pump containing Dow Corning 705 diffusion pump oil. A Freon cooled baffle prevents migration of oil into the system. Basic pressure in the accelerator is about 10^{-6} torr as measured by a Phillips ionization gauge and pressure during operation is in the $0.6 - 1 \times 10^{-5}$ torr range.

The accelerator is equipped with an analyzing magnet to provide mass analysis of the ion beams. The analyzed beam which is used is bent at an angle of 45° to the accelerator axis.

D. Transition Section and Beam Scanner

After leaving the analyzing magnet, (Item 7, Figure 1), the beam passes through a glass cross (Item 9, Figure 2). Within the cross a monitor, consisting of a Vycor disc covered with a stainless steel screen, can be positioned to intercept the beam. This provides a beam current readout at that point and also a visual observation, since the glass fluoresces when the ions or electrons impinge upon it.

The accelerator and chamber are separated by a differentially pumped section to isolate the ultra-high vacuum region of the chamber from the moderately high vacuum region of the accelerator. The differentially pumped section is terminated at either end by rectangular slit orifices which help to limit gas conductance, and also serve to collimate the charged particle beam prior to entrance into the beam scanner. Pressure in the differential section is normally about 1.5 orders of magnitude lower than that in the accelerator. Pressures in the chamber are normally in the 10^{-8} to 10^{-10} torr range during operation.

The beam scanner consists of a pair of electrostatic deflection plates. The collimated beam passes between these plates and is moved to sample center by application of a d.c. bias and then "rastered" across the required area by means of a low frequency sawtooth voltage applied to the plates.

E. Solar Simulator

The solar simulator utilizes a 5 kilowatt short-arc lamp, either xenon or xenon-mercury to simulate the solar spectrum. The light from the simulator is beamed through a quartz window in the port (Item 14, Figure 1), and reflected from an aluminized front face mirror onto the samples. The lens system in the simulator provides for focusing the light such that with the end of the simulator barrel about one-fourth inch from the quartz window flange, a uniform three-inch diameter beam is obtained at a distance of 14 inches from the mirror. Total intensity was expected to be in excess of ten solar constants with either type tube. However, it was not possible to achieve ten solar equivalents in the 0.2 - 0.4 micron range with the Xe lamp so that the Xe-Hg lamp was used for the balance of the study.

F. Lyman-Alpha Source

The vacuum ultraviolet source of Avco design is essentially a Penning discharge tube with water-cooled anode and cathodes. Light from this source is beamed through a lithium fluoride window onto the samples. The source provides energy in the 0.105 to 0.2 micron range. With hydrogen gas it is essentially a line source with the Lyman-alpha line at 1216 Å as the most prominent. The port for the source is shown in Figure 1, Item 13.

G. Reflectance Measuring Apparatus

Reflectance measurements are made with a Perkin-Elmer Model 112-U spectrophotometer attached to the Avco designed integrating sphere. A-Pek Model X-75 xenon lamp and Gier-Dunkle Model RXS-1 power supply were obtained to provide sufficient intensity for reflectance measurements below 0.4 microns. A tungsten lamp is used for measurements at longer wavelengths.

H. Non-In Situ System

The system used for irradiation and measurement of samples for <u>non-in situ</u> testing has been used in performance of other contracts and is adequately described in previous reports. (1)

IV. EQUIPMENT CALIBRATION AND EVALUATION

A. In Situ Reflectometer System

Evaluation of the in situ reflectometer was performed by comparing spectral reflectance measurements in the in situ integrating sphere with measurements in the Gier-Dunkle AIS-6 integrating sphere for a ZnO/K2SiO3 sample and for SiO on vapor deposited aluminum. Five sets of spectral reflectance measurements were made on each sample to check repeatability of the equipment. The attached tables show the measured values obtained in the Gier-Dunkle sphere and in each of the five sets of measurements in the in situ sphere, the averages of the five sets of measurements, and the maximum deviation from the average at each wavelength. Measurements are presented only over the wavelength range measureable with a tungsten lamp because of problems with the xenon lamp power supply.

Figures 7 and 8 present comparative spectral reflectance curves for the two spheres for the ZnO/K₂SiO₃ and SiO_x on vapor deposited aluminum respectfully.

B. Particle Energy and Flux

Particle energy is essentially determined by the potential drop across the acceleration tube in the accelerator. When the Van de Graaff voltage generator is being used, the generating voltmeter on the Van de Graaff has been calibrated to read directly the particle energy. The calibration was performed as follows:

The particle beam from the accelerator after collimation by the orifice slits passes between the electrostatic deflection plates of the beam scanner. The beam was permitted to fall on a Pyrex window and the resultant fluorescense marked the position of the beam. By applying a d.c. potential to the deflection plates the beam was moved to a different position. The accelerating potential was determined from the well known equation

$$V_A = \frac{1d}{2tx} Vd$$

TABLE II

Comparison of Measurements in In Situ Sphere With Those in Gier-Dunkle Sphere at Atmosphere

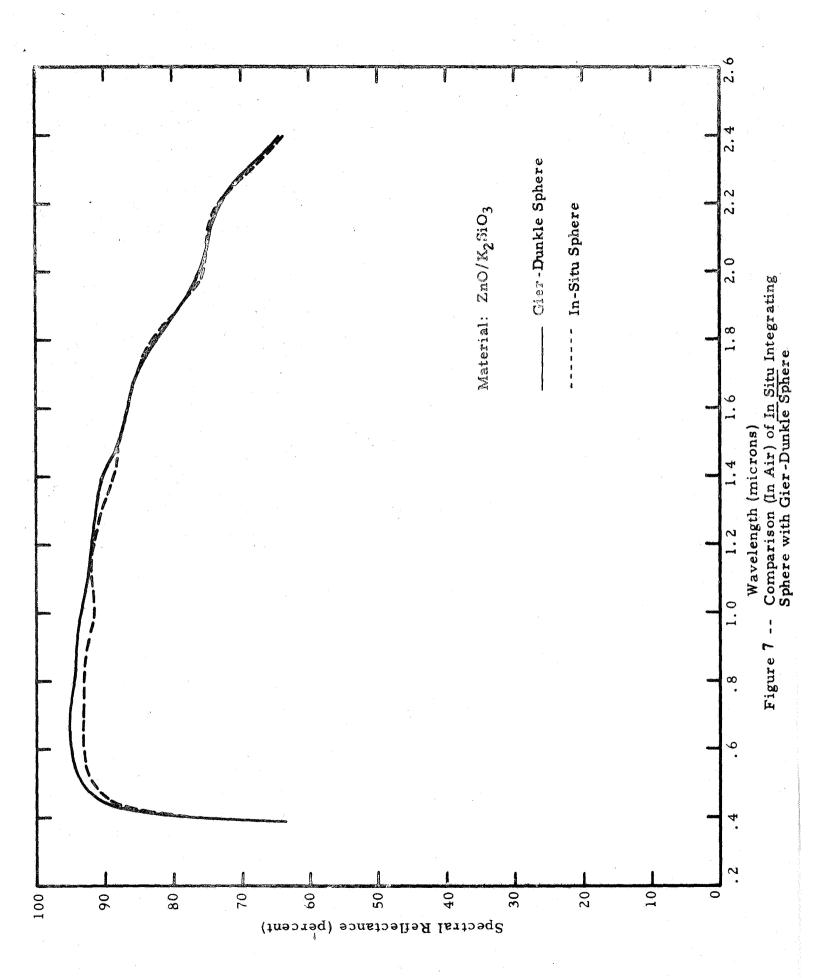
for ZnO/K2SiO3

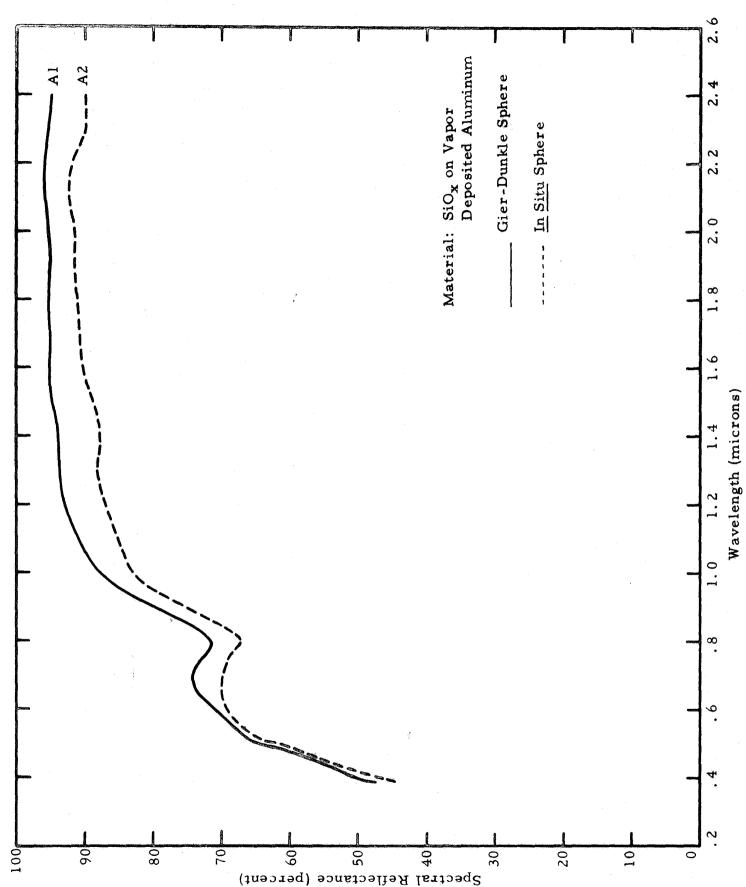
		2	pectral	Reflect	ance			
Wavelength	Gier-	_		In Situ S		_		Max. Deviation
(Millimicrons)	<u>Dunkle</u>	1		3	4	5	Ave.	From Average
390	63.5	63.4	63.0	64.6	64.3	62.9	63.6	1.0
400	78.4	77.1	75.7	76.4	76.2	76.5	76.4	0.7
410	82.6	81.7	81.7	81.6	80.7	81.5	81.4	0.7
420 430	86.0 88.0	85.5	84.4	84.4	81.7	85.1	8 4.2 87.0	1.3
440	89.8	85.8 88.2	86.3 87.7	86.4 89.3	89.1 88.4	87.2 87.7	88.3	2.1
450	90.6	89.7	89.2	89.4	88.1	89.3	89.1	1.0
460	91.8	90.4	89.6	90.1	91.2	89.7	90.2	1.0
470	92.1	91.0	89.9	90.4	90.8	90.0	90.4	0.6
480	92.5	90.3	90.3	90.7	91.2	90.7		• •
490	93.6	91.1	91.7	91.9	90.9	91.5	91.4	ω , 0.6 Ω 0.5
500	94.6	91.2	91.5	92.0	91.9	91.6		0.4
510	93.8	92.4	91.8	92.8	93.3	92.2	,	_: 0.8
5 2 0	94.1	93.1	93.2	93.5	92.6	93.1	93.1	. 0.8 ≥ 0.5
530	94.5	91.7	93.5	93.0	91.8	93.2		ը <mark>՝</mark> 0.9
540	94.5	92.8	92.1	92.8	92.7	93.8	92.8	1.0
550 510	94.3	91.3	92.0	93.5	93.2	93.5	92.7	1.4
560	94.6	91.7	93.2	93.8	92.3	93.6	92.9	1.2
570	94.6	92.8	93.5	93.2	93.7	93.9	93.4	0.6
580 600	94.8 94.4	93.2 93.3	93.9 92.9	92.5 93.3	93.8 92.7	94.1 93.7	93.3 93.2	0.8
630	94.8	93.4	93.8	94.3	93.9	94.7	94.0	0.7
660	95.3	92.5	93.5	93.7	94.4	92.9	93.4	1.0
600	95.9	92.2	92.4	93.2	92.8	93.4	92.8	0.6
630	95.1	92.4	91.9	92.5	93.5	93.0	92.7	0.8
660	95.1	92.4	92.1	92.9	92.3	93.4	92.6	7 0.8
700	95.3	93.4	92.4	92.4	93.2	94.1	93.1	1.0
75 0 800	94.9	92.9	92.8	93.7	92.7	93.2	93.1	0.6
800 900	94.4 94.1	93.1 92.0	92.3 92.4	93.6	92.3	93.9	93.0	0.9
1000	93.4	89.6	92.4	93.1 93.5	92.4 91.2	92.9 91.7	92.6 91.6	0.6
1100	92.5	91.7	91.7	92.2	91.8	92.3	91.9	0.4
1200	91.8	92.3	91.0	91.7	91.9	90.5	~ 7 =	• • • • • •
1300	91.2	90.4	91.2	90.0	90.1	90.4	90.4	0.8
1400	90.3	89.2	88.5	88.9	88.6	88.5		0.5
1500	87.9	87.5	88.2	88.3	88.4	87.7		ο. 5
1600	86.7	86.7	87.0	86.4	87.4	86.2	86.7	瓦 0.7
1700	85.2	84.9	85.0	85.2	85.9	85.5	85.3	0.6
1800 1900	82.5 78.9	82.5	83.0	83.4	82.7 78.7	83.7	83.1	0.6
2000	75.7	78.9 75.4	79.4 75.4	79.1 75.8	76.3	79.5 75.2	79.1 75.6	0.4
2100	74.6	74.8	74.9	74.9	74.4	75.8	75.0	0.8
2200	73.0	73.2	73.3	73.2	73.5	74.1	73.5	0.6
2300	68.8 64.2	68.6	68.7 63.7	68.7 63.9	68.3 63.4	68.8 63.8	68,6 63.8	0.3
2400	64.2	64.1	63.7	63.9	63.4	63.8	63.8	0.4

TABLE III

Comparison of Measurements in In Situ Sphere With Those in GierDunkle Sphere at Atmosphere for SiO_V on Vapor Deposited Aluminum

Spectral Reflectance In Situ Sphere Wavelength Gier-Max. Deviation 2 (Millimicrons) Dunkle 1 4 5 From Average Ave. 0.7 390 47.5 45.1 43.9 44.1 44.4 44.4 44.6 400 50.2 46.2 45.3 46.6 46.2 46.2 46.1 0.8 410 51.4 47.2 47,9 47.9 48.2 47.8 0.6 48.0 420 52.9 49.5 49.9 50.4 49.6 50.2 49.9 0.5 430 52.9 1.5 51.8 52.0 51.5 51.1 53.5 52.0 54.7 52.9 0.5 440 53.5 53.6 53.3 53.7 53.4 0.7 54.0 55.2 55.2 54.7 450 56.4 54.7 54.5 460 57.8 55.8 55.8 56.4 56.5 56.5 56.2 0.4 56.9 470 58.8 56.4 57.2 57.3 57.1 0.7 57.6 Tube 480 60.5 57.5 59.0 58.6 58.5 59.1 58.8 1.1 62.2 490 60.0 59.9 60.5 60.3 60.1 60.2 0.3 500 64.6 61.2 61.9 61.8 61.4 62.3 61.7 0.5 510 66.3 63.0 63.2 63.3 63.3 63.7 63.3 0.4 64.6 65.5 66.6 520 64.4 65.0 64.4 64.8 64.4 0.4 Ω_{i} 0.7 530 66.8 65.3 64.8 65.4 66.0 65.8 67.9 0.7 540 65.4 66.7 66.0 65.8 66.6 66.1 68.1 550 66.4 67.0 67.1 66.8 0.4 66.6 66.7 560 68.9 67.5 0.7 67.5 68.2 67.0 67.7 66.9 570 69.4 67.5 68.0 68.1 67.8 68.5 68.0 0.5 0.7 70.2 68.9 580 68.4 69.5 68.8 68.6 68.8 68.7 69.1 70.3 600 71.1 69.2 68.6 69.8 69.1 0.7 630 72.5 69.6 70.0 70.3 70.1 70.1 0.5 73.7 660 69.7 71.3 70.7 70.4 71.1 70.6 0.9 600 71.3 67.2 69.2 67.3 67.4 68.2 1.3 67.9 72.3 73.8 630 67.9 69.1 69.2 68.6 69.7 68.7 70.9 68.6 0.7 68.7 660 $70.\bar{2}$ 69.1 69.8 1.1 74.3 700 69.3 70.0 70.1 69.4 70.3 69.8 0.5 69.0 750 72.9 68.5 69.7 69.2 69.2 68.4 0.7 67.2 75.6 66.5 75.6 67.3 67.4 75.8 67.4 75.6 67.3 75.2 800 71.4 0.7 900 75.6 82.9 0.4 80.0 82.3 1000 87.9 83.2 82.6 83.5 82.9 0.6 90,9 85.0 1100 84.0 84.9 85.9 85.0 85.4 0.9 93.0 93.8 87.8 89.0 86.4 87.8 87.0 88.3 1.2 0.7 1200 85.8 88.7 87.7 88.1 87.4 87.8 eII 1300 Ũ 87.9 93.9 0.7 1400 88.0 88.4 88.4 87.4 87.2 S 94.8 1500 88.0 89.0 88.9 89.4 88.8 88.8 0.8 D D 1600 95.3 89.8 90.2 90.8 89.8 90.3 90.2 0.6 95.2 90.9 1700 90.7 91.5 91.0 90.5 90.8 0.6 91.2 1800 95.4 90.1 91.0 91.7 91.4 91.6 1.1 1900 95.3 91.0 91.8 92.0 91.9 91.5 91.6 0.6 92.0 91.9 91.9 2000 95.4 90.5 92.0 91.7 1.2 92.9 92.4 92.4 92.0 2100 95.8 91.8 93.1 92.2 92.2 0.9 2200 2300 95.9 95.5 92.2 89.5 92.4 91.3 91.7 0.7 89.9 90.6 90.0 90.1 89.8 0.6 94.9 0.4 2400 89.8 90.4 90.1 89.8 90.0 90.0





Comparison (In Air) of In Situ Integrating Sphere with Gier-Dunkle Sphere Figure 8

where V_A is the accleration voltage, 1 is the length of the deflection plates, d is the distance from the center of the plates to the glass window, V_d is the applied deflection voltage, t is the spacing between the plates, and x is the distance the beam was moved on the window.

The calibration was made at several accelerating potentials to accurately calibrate the readout meter.

When the auxiliary power supply is used, the reading from the meter on its control panel is used to determine the particle energy, i.e., a 10 kilovolt potential produces 10 Kev particles. This is not strictly true in the case of positive ions since in addition to the potential across the acceleration tube, the ions are given an initial acceleration to extract them from the R.F. ion source by application of a relatively low potential to the source probe. However, the total energy of the particles is known to within ten percent.

Charged particle flux is readily determined due to the geometry of the sample holder. As described in Section III. A. 2, the collimating plate mounted in front of the sample holder defines the area, A, of the sample and sample holder which is irradiated. The average current striking this area is directly measured by connecting a meter from the sample holder to ground. If this current, I, is noted in amperes, the flux is given by

$$\frac{I}{A}$$
 x 6.25 x 10¹⁸, particles/cm²/sec

When a certain flux is required it is, therefore, simple to determine the required beam current and adjust the accelerator accordingly. The method used in adjusting for the desired current is to adjust for a current on the monitor in the glass cross section which will yield approximately the proper target current and to make final minor corrections after the beam is on target.

The target current is recorded on a strip chart recorder throughout the tests. The current is sufficiently stable that the total integrated flux in particles/cm² striking the samples during a test is known within an estimated fifteen percent.

C. Solar Simulator Calibration

The solar simulator was calibrated by duplicating the vacuum chamber geometry on a work bench. Spectral calibrations were made using a monochromator calibrated with an NBS standard quartz-iodine lamp, thus providing quantitative spectral intensity data. The ellipsoidal collecting mirror which was provided for the simulator was coated with an aluminized coating. Figure 9 compares plots of simulator spectral intensity with a Hg Xe lamp and using the aluminized collector with a plot of solar extraterrestrial spectral intensity according to Johnson. An analysis of the curve for the aluminized collector in Figure 9 shows that with 10 solar equivalents below 0.4 microns there were approximately 2.5 equivalents between 0.26 and 0.28, and 15 equivalents in the 0.26 to 0.30 micron range. These measurements were made with a new Xe-Hg lamp in the simulator.

D. Vacuum Ultraviolet Calibration

The vacuum ultraviolet light calibration was made with one centimeter nickel discs mounted at the sample positions in the sample holder, and with a nitric oxide ionization cell with a lithium fluoride window also at sample position. The nickel discs serve as photoelectric detectors. With a negative bias applied to the discs, the photoelectrons, which are generated primarily by Lyman-alpha photons, are ejected and the current is proportional to the light intensity. A quantum efficiency of 2.5 percent was assumed for the nickel based on data of Hinteregger and Watanabe. The overall quantum efficiency of the NO cell obtained from G.B.L. Associates was not known. Dunkleman (10) states that efficiencies range from 10 to 50% for similar detectors.

A loop of 10 mil nickel wire permanently mounted close to the light source was cross calibrated to provide continuous monitoring of light intensity during the tests.

E. Non-In Situ System

Calibration of this system is adequately described in previous reports. (1)

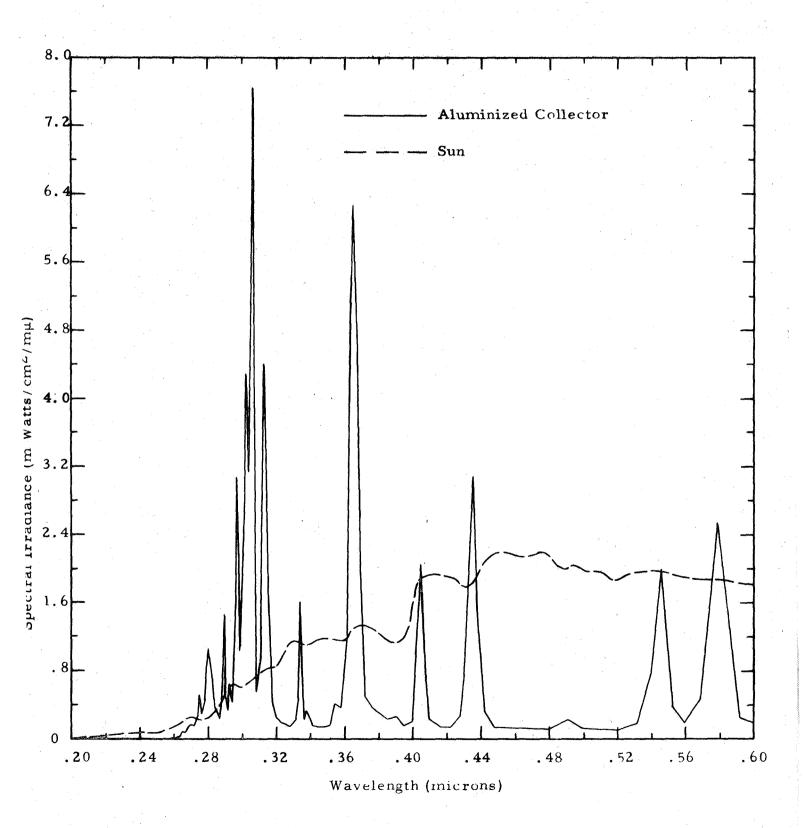


Figure 9 - SPECTRAL IRRADIANCE OF SOLAR SIMULATOR AT 10 SOLAR EQUIVALENTS

V. TEST PARAMETERS AND RESULTS

A compilation of all the sample materials, environmental parameters and values of α_s and $\Delta \alpha_s$ of the in situ tests are included in Table IV. Two values of $\Delta \alpha_s$ are presented, $\Delta \alpha_{s_l}$ is the change at the end of the accelerated radiation, and $\Delta \alpha_s$ is the change at the end of the test. The proton energy for each test was 10 Kev, the sample temperature was maintained at room temperature and the chamber pressure was 10^{-7} torr or less for every test. These items are not tabulated. Except in the tests with the xenon lamp which is given the near and vacuum, ultraviolet was applied at a rate of 10 solar equivalents for a total of 750 sun hours of electromagnetic radiation. This was chosen because it had been previously shown in non-in situ testing that this was about the minimum necessary to cause measurable optical damage to ZnO_2/K_2SiO_3 samples.

A total integrated proton flux of 2 x 10¹⁵ p/cm² was selected in the majority of the tests. In one test a total integrated flux of 1 x 10¹⁶ p/cm² was selected in order to determine if additional proton damage could be detected. In order to determine if a rate effect existed, some tests were conducted at an accelerated rate approximately 100 times the continuous rate. In the accelerated tests, the flux was applied for approximately one hour in order to obtain the same total integrated flux as in the continuous irradiation tests. Reflectance measurements were made on the samples in situ and were made at various times during the irradiation. The reflectance measurements were made over the range .34 to 2.4 microns.

TABLE IV

	Initial Δa_{s_1} Δa_{s_2}	$\begin{array}{cccccccccccccccccccccccccccccccccccc$	5 . 190 . 046 . 041 5 . 179 . 043 . 035	5 . 192	5 .287020 5 .287 .027 .035 5 .291 .035 .031	5 .287043 5 .286048
S	Protons	5.3 × 10	2 x 1015 2 x 1015	2 x 10 ¹⁵ 2 x 10 ¹⁵	2 x 1015 2 x 1015	2×10^{15} 2×10^{15}
TEST PARAMETERS AND RESULTS	p/cm ² sec	1.1 x 1012 1.1 x 1012	5.5 x 1011 5.5 x 10	3.2 × 109 3.2 × 109	5.2 x 1011 5.2 x 1011	7.35 x 10 ⁹
ARAMETERS	Lyman-a Sun Hrs.		•		750	750
TEST P.	Near UV Sun Hrs.				750	750
አ ያ	Sample No.	1-11-9-13 #5 1-11-9-13 #6 1-11-9-13 #7 1-11-9-13 #8	1-11-9-13 #9 1-11-9-13 #10 1-11-9-13 #11 1-11-9-13 #12	1-11-9-13 #13 1-11-9-13 #14 1-11-9-13 #15 1-11-9-13 #16	F-1-47-D #1 F-1-47-D #2 F-1-47-D #3 F-1-47-D #4	F-1-47-D #5 F-1-47-D #6 F-1-47-D #7
	Material	$ m ZnO/K_2SiO_3$	$ m ZnO/K_2SiO_3$	$ m ZnO/K_2SiO_3$	Hg Xe Lamp $4 \text{ZnO/K}_2\text{SiO}_3$	$ m ZnO/K_2SiO_3$
	Test No.	П	8	en .	Hg Xe	ία ·

-24-

$\Delta \alpha_s$	036 025 002	. 021 . 083 . 047	. 016 . 044 . 030	980 980 900	012	. 012	. 017
$\Delta \alpha_s \Delta \alpha_$	9003		. 028	. 010	1 1 1 1 1 1 1 1 1 1 1 1 1 1 1 1 1 1 1	1	t 1 1 1 1 1 1 1 1 1 1 1 3 3 3 1
Initial a	.373	.210 .214 .217	.213 .211 .211	. 201 . 194 . 194	. 189	. 224	.212
s p/cm ²	2×10^{15} 2×10^{15} 2×10^{15}	2×10^{15} 2×10^{15}	2×10^{15} 2×10^{15}	2×10^{15} 2×10^{15} 2×10^{15}	1×10^{16} 1×10^{16}	2×10^{15} 2×10^{15}	3×10^{15} 3×10^{15} 3×10
Protons	2×10^{11}	.35 × 109	.5 x 10 ¹¹ .5 x 10 ¹¹	$.2 \times 10^{11} \\ .2 \times 10^{11} \\ .2 \times 10^{11}$	5×10^{11} 5×10^{11}	.35 x 109	5 x 1011
Lyman-a Sun Hrs. p	750 750 5	750 7750 7750 7750 7750 7750 7750 7750	750	750 750 55	; ; ; ; ; ; ; ; ; ; ; ; ; ; ; ; ; ; ;	750 7 750 7	190
Near UV Lym Sun Hrs. Sun	750	750	750	750 750 	1 1 1 1 1 1	750	190
Batch & Sample No.	F-1-53-A #1 F-1-53-A #2 F-1-53-A #3 F-1-53-A #4	F-1-55 #1 F-1-55 #2 F-1-55 #3 F-1-55 #4	F-1-55 #5 F-1-55 #6 F-1-55 #7 F-1-55 #8	F-1-38#1 F-1638#2 F-1-38#3 F-1-38#4	F-1-38 #5 F-1-55 #9	A275 #1 A275 #2 A275 #3 A275 #4	F-1-56 #1 F-1-56 #2 F-1-56 #3 F-1-56 #4
Material	$\text{La}_2\text{O}_3/ ext{K}_2 ext{S}_1\text{O}_3$	ZnO/Silicone	ZnO/Silicone	${ m La_2O_3/K_2SiO_3}$	La ₂ O ₃ /K ₂ SiO ₃ ZnO/Silicone ³	S-13G	TiO ₂ /Silicone
Test No.	9	2	8	I 6	10 I	111 S	12 1

								•	
Act Act S	. 108	. 004	. 074 . 058	. 077 . 046		δα s	. 035	. 003	. 000
Initial as	. 158	. 108	. 184	. 114		Initial as	. 124	. 120 . 125 . 124	. 064
$\frac{1}{2}$	5×10^{15} 5×10^{15}	5×10^{15} 5×10^{15}	5×10^{15}	5×10^{15} 5×10^{15}		$\frac{s}{p/cm^2}$	2×10^{15}	2 x 10 ¹⁵	1.5 x 10 ¹⁷
Protons p/cm ² /sec	5.5×10^{11} 5.5×10^{11}	5.5×10^{11} 5.5×10^{11}	5.5×10^{11}	5.5×10^{11} 5.5×10^{11}	TESTS	Protons p/cm ² /sec	5.5×10^{11}	7.4 × 10 ⁹	5.5 x 10 ¹¹
Lyman-a Sun Hrs.	1 T t .1 t t		1 2 3	1	NON-IN SITU TESTS	Lyman-α Sun Hrs.	1 1 1	750	750
Near UV Sun Hrs.	1 1 1 1 1 1	f 1 1 1 1 1	417	1 1 1 1 1		Near UV Sun Hrs.	! ! !	750	750
Batch & Sample No.		(E	1-11-9-13 #17	F-1-47-D #9		Batch & Sample No.	1 1 1 1 1 1	\$ 1 1 1 1 1 2 3 4 4 5 5 1 4 5 1 1 1 2 1 1	1 1 1 1 1 1 1 1 1 1 1 1 1 1 1 1 1 1 1
Material	MgO (powdered) TiO ₂ (powdered)	SiO_2 (powdered)AL $_2O_3$ (powdered)	$\mathrm{ZnO/K_2SiO_3}$	LaO_3 (powdered)ZnO/ K_2SiO_3 F-1-47-D #		Material	ZnO Powder	ZnO powder	Vac. Dep. Ag on Fused Silica
Test No.	13	4.	12	16		Test No.	-4	7	m

VI. DISCUSSION OF RESULTS

A. In Situ Tests

Sixteen exposure tests were conducted on various samples in which reflectance measurements were made in situ. Four samples were included in each test.

- 1. A sample exposed to vacuum, controlled temperature, and solar simulation.
- 2. A sample exposed to vacuum, controlled temperature, solar simulation and protons.
- 3. A sample exposed to vacuum, controlled temperature and protons.
- 4. A control sample exposed to vacuum and controlled temperature.

Proton exposures were conducted with a pure beam of 10 Kev protons.

1. Xenon Lamp

Three exposure tests were conducted on ZnO/K₂SiO₃ samples in which reflectance measurements were made in situ. Solar simulation for the tests was provided by a simulator using a 5 kilowatt xenon lamp. Spectral irradiance was to have been at a level of 10 solar equivalents of near ultraviolet. However, this level could not be attained so the highest levels attainable were used. Proton exposures were conducted with a pure beam of 10 Kev protons. All tests were conducted at a nominal temperature of 298°K as measured by a thermocouple in contact with the sample rack.

For the first test, simulated solar irradiance at sample position was approximately 5.5 solar constants with 3 solar equivalents in the near ultraviolet. Proton flux was greater than planned because of an error in current readout and because the beam narrowly missed one edge of the samples due to a misalignment in the beam line.

Proton flux was approximately 1.1×10^{12} p/cm²/sec and the integrated flux was 5.3×10^{15} p/cm². Because of the error in the proton flux, the test was stopped immediately after the proton irradiation.

Figures 10 and 11 show the change in spectral absorptance for the sample irradiated only with protons and for the sample exposed to the combined environment. The change in solar absorptance was .082 for the former and .063 for the latter. Changes are based upon pre-exposure measurements made in vacuum.

For the second test spectral reflectance measurements were made on each of the four samples before evacuating the test chamber and again about 24 hours later at a pressure in the 10⁻⁹ torr range. The primary result of the vacuum environment was to increase the reflectance of the samples by approximately 2% in the wavelength range of 1.6 to 2.4 microns.

The solar irradiance was the same at the beginning of this test as in the first test. The proton flux was to have been $5.5 \times 10^{11} \text{ p/cm}^2/\text{sec}$ for one hour to obtain an integrated flux of $2 \times 10^{15} \text{ p/cm}^2$. However, a problem in the proton beam scanning supply caused the beam to miss the sample exposed only to protons for a part of the irradiation period. After stopping the test it was found that the aluminum surface had evaporated from the mirror which reflects the light from the solar simulator onto the samples. This mirror is located in the vacuum chamber and could not adequately dissipate the heat from the absorbed infrared radiation.

Figure 12 is plotted from data taken for the sample exposed to the combined environment, since this sample was uniformly irradiated even though the proton flux and solar exposure cannot be accurately evaluated. The curves in Figure 12 are based upon changes from the pre-exposure spectral reflectance measurements in vacuum. The solid curve shows the change in spectral absorptance induced by the radiation from data taken immediately following proton irradiation. An absorption

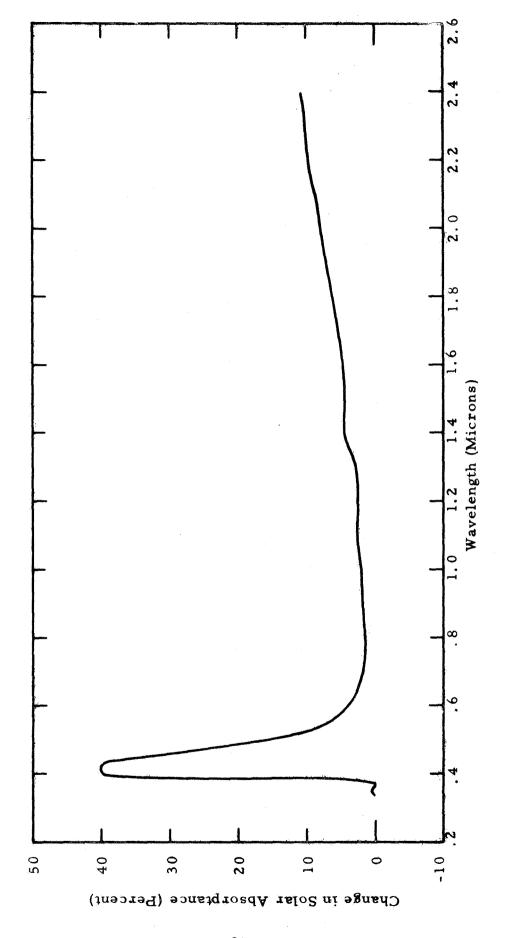
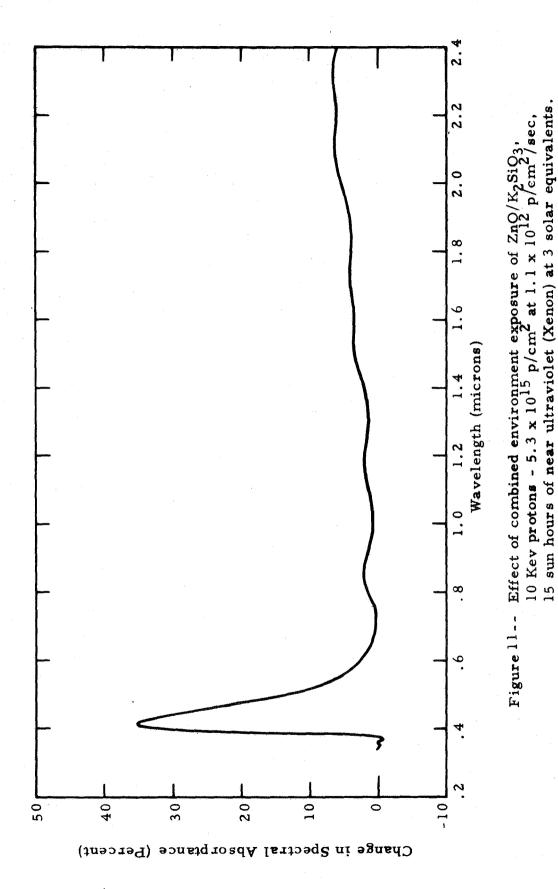
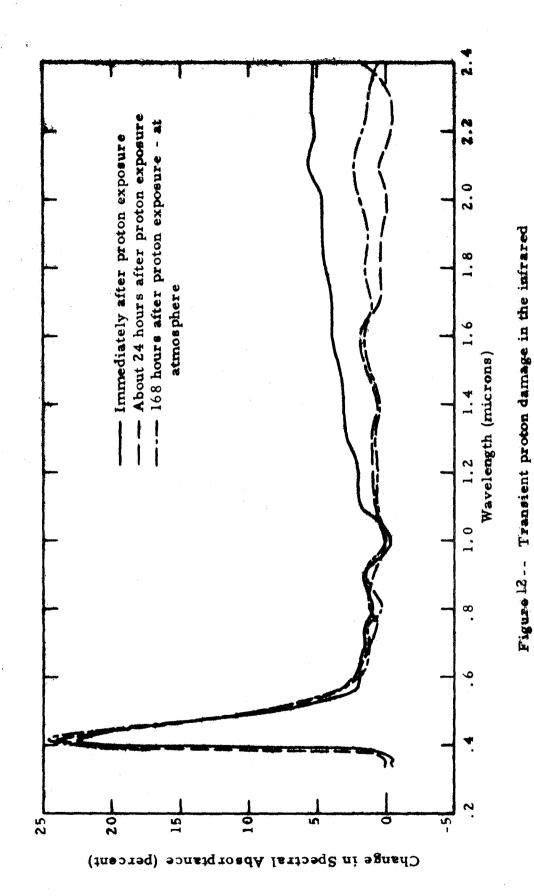


Figure 10-- Effect of 10 Kev protons on ZnO/K₂SiO₃ - 5.3 x 10¹⁵ p/cm² at 1.1 x 10¹² p/cm²/sec



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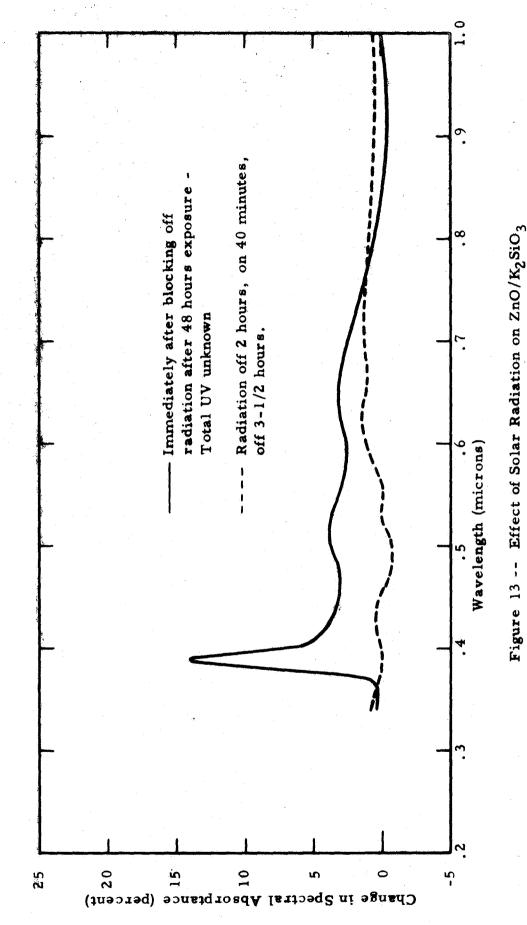
ZnO/K2SiO3

band was induced from .31 to .55 micron and another above 1 micron. The data taken about 24 hours later shows a recovery of infrared damage even though chamber pressure was less than 10⁻⁹ torr. The recovery is not an effect of the solar radiation since the sample which was exposed only to protons exhibited the same recovery; nor was it a continued effect of vacuum since the control sample did not exhibit any further change following the initial measurements in vacuum. The other curve shows an increase in infrared reflectance following return of the chamber to atmospheric pressure.

Pre-exposure spectral reflectance measurements were made both in air and vacuum for the samples used in the third test and they exhibited the same increase in reflectance in the infrared as had the previous group.

The test was set up for a proton flux of $3.2 \times 10^9 \text{ p/cm}^2/\text{sec}$ and an integrated flux of $2 \times 10^{15} \text{ p/cm}^2$. With a new mirror with a somewhat improved heat sink installed in the chamber the solar irradiance at sample position as measured with a pyrheliometer was 7.5 solar constants with 4.3 solar equivalents in the near ultraviolet. When the test was stopped after what should have been 750 sun hours of ultraviolet exposure, it was found that the aluminum had evaporated off the new mirror.

Figure 13 shows an effect of the solar radiation. The solid curve is from spectral reflectance measurements made immediately after shutting the radiation off the samples after approximately 48 hours of exposure. The sample was exposed only to solar radiation. The dashed curve is from measurements made when the radiation had been off for 2 hours, on for 40 minutes, and then off for 3-1/2 hours. The radiation apparently caused a decrease in reflectance primarily around the absorption edge but in a short period of time after blocking off the radiation the absorption peak disappeared. At the end of the test, this sample exhibited considerable more optical damage than would have been expected even from a full 750 sun hours of near ultraviolet. A check showed that the samples



were not seating properly in the sample holders. Thermal transfer was probably inadequate to keep the samples exposed to the simulated solar radiation from heating. The transient absorption peak was an anomalous result and was never reproduced.

The sample which was exposed to the combined environment suffered more damage than the sample exposed to protons. This was in contradiction to the results of previous tests of ZnO/K₂SiO₃ when the samples were maintained at the same temperature and is a further indication that the samples exposed to the solar radiation may have been considerably warmer than the other two samples.

Figures 14 and 15 show changes in spectral absorp tance caused by proton exposure and by combined proton and solar radiation exposure respectively. The three curves represent the change which had occurred at various stages of exposure. Changes in solar absorptance are noted on the figures.

2. Mercury Xenon Lamp

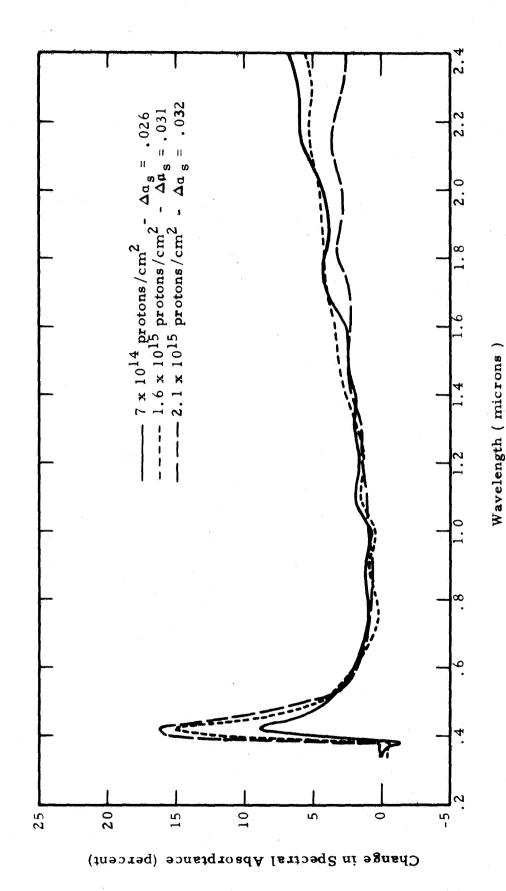
Thirteen exposures were conducted on the various materials using a 5KW Mercury Xenon Lamp in the solar simulator. The results of these tests are discussed in the following paragraphs based upon the environmental parameters selected. The curves which accompany show only changes in spectral absorptance.

a. Zinc Oxide/Potassium Silicate

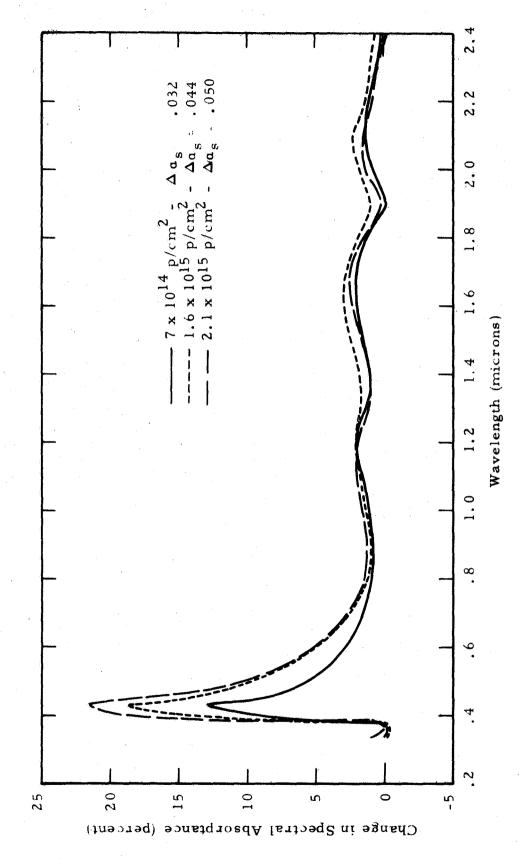
Two batches of zinc oxide potassium silicate were tested, F-1-47-D and 1-11-9-13. Effects of the individual irradiations as well as the combined environment are discussed below.

1. Proton Effects

The effects of proton radiation only are shown in Figures 16 and 17 for the F-1-47-D ZnO/K₂SiO₃ and Figure 18



Effect of 10 KEV protons on $\rm ZnO/K_2SiO_3$ at $\rm 298^{\rm o}K$ Average proton flux 3.8 x 10^9 p/cm²/sec Figure 14 --



Effect of Combined Environment on ZnO/K2SiO₃ at 298°K Average proton flux 3.8 x 10⁹ p/cm²/sec - Ultraxiolet unknown. Figure 15 --

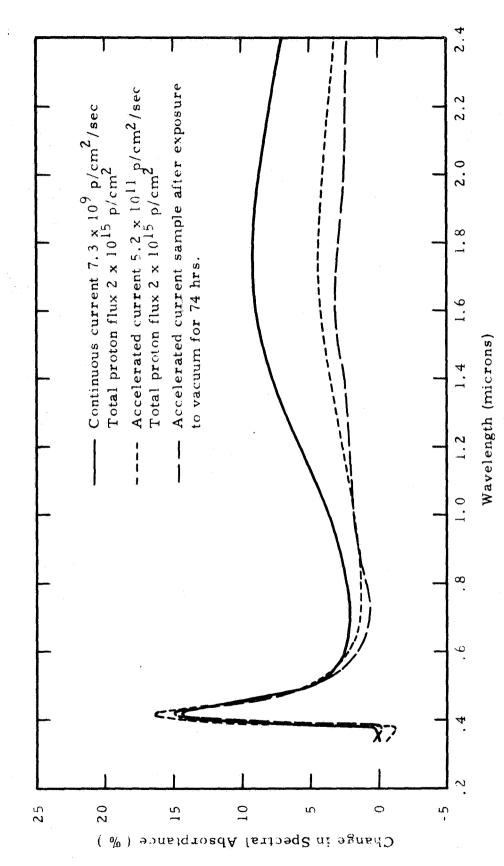
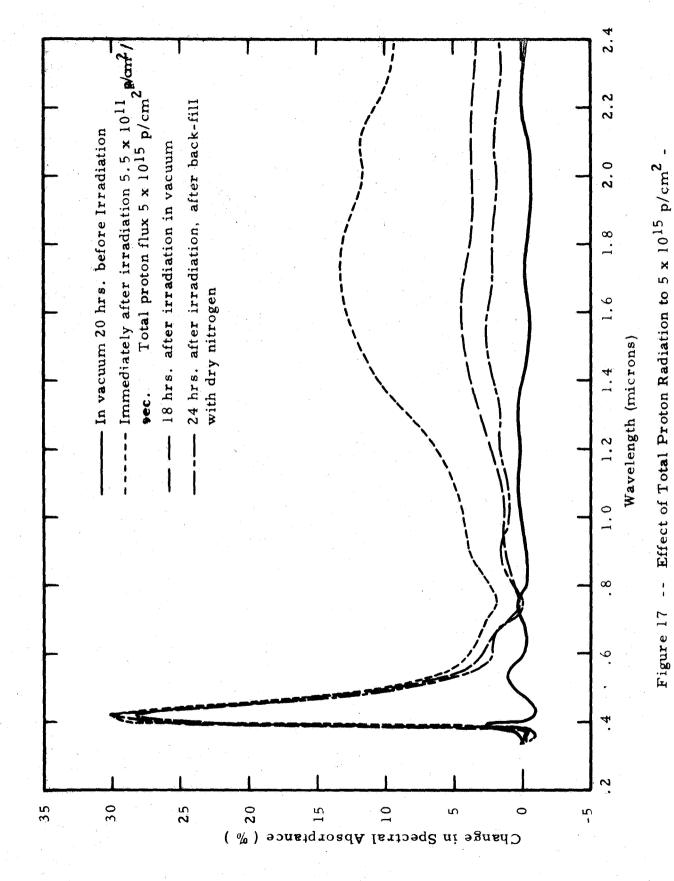


Figure 16 -- Rate and Vacuum effect of proton radiation only - ZnO/K_2SiO_3 - (F-1-47-D)



ZnO/K2SiO3 - (F-1-47-D)

shows the effect on the 1-11-9-13 ZnO/K₂SiO₂. Greater damage is observed in the infrared region due to the continuous proton radiation than with the accelerated flux indicating a rate effect. Also shown in Figure 16 is a slight bleaching effect in the infrared region for the sample which was irradiated at the beginning of the test and remained in vacuum while the other samples were irradiated with ultraviolet. Figure 17 also shows this effect. In Figure 17, the effect of increasing the proton flux 2-1/2 times is shown and allowing the sample to remain in vacuum for a specified period of time.

Figure 18 shows the effect of proton radiation only to the 1-11-9-13 material and again remaining in vacuum after irradiation. Again, the bleaching effect may be noted.

2. Ultraviolet Damage

Figure 19 shows the effect of ultraviolet radiation on the F-1-47-D ZnO/K₂SiO₃ material. In both tests the ultraviolet was applied at a rate of 10 suns. The damage was less than 5% in the near ultraviolet, visible and infrared region as may be seen from this figure. Results were essentially the same for two separate runs on this material.

3. Combined Effects

The combined effects of proton, vacuum and ultraviolet environment are shown in Figures 20, 21, 22, and 23.

Figures 20 and 21 show a comparison between the combined radiations on one

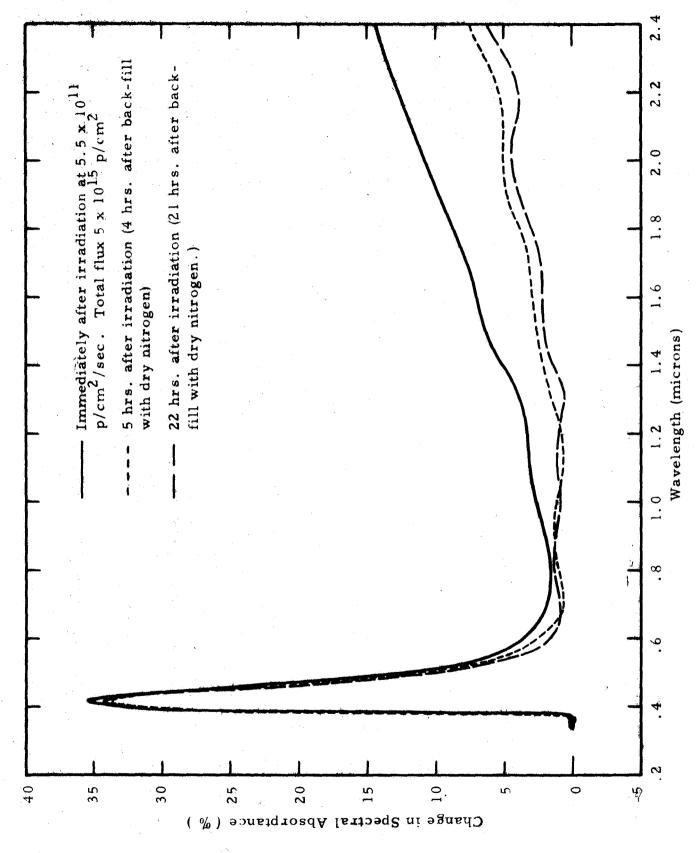
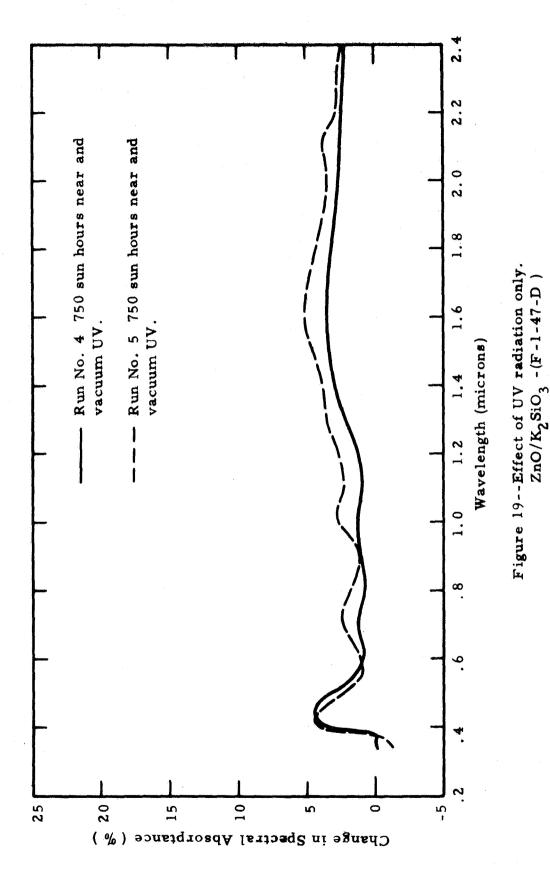
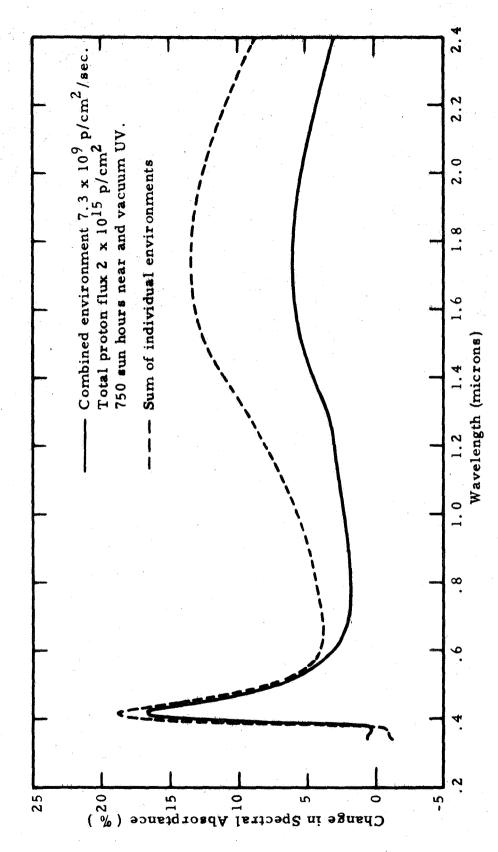


Figure 18--Effect of protons on 1-11-9-13 ZnO/K₂SiO₃, Remaining in vacuum after irradiation





Combined effects versus sum of individual effects. Continuous low current - ZnO/K2SiO3 (F-1-47-D) Figure 20 --

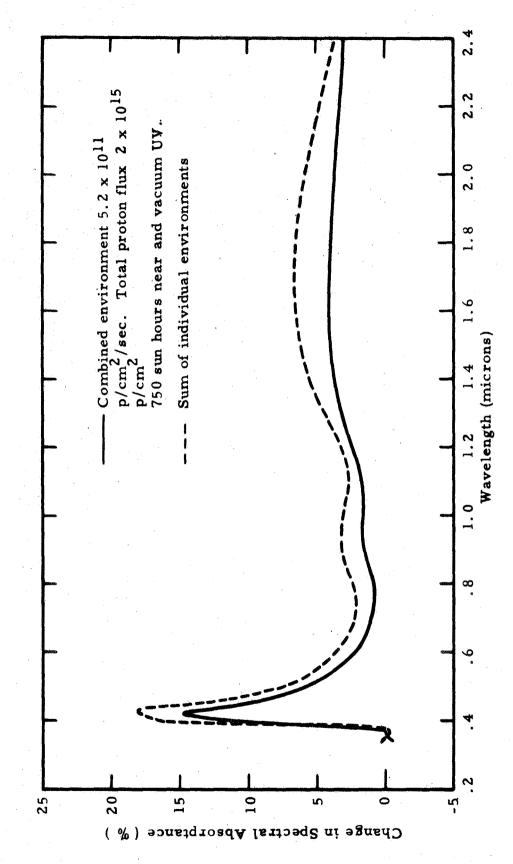
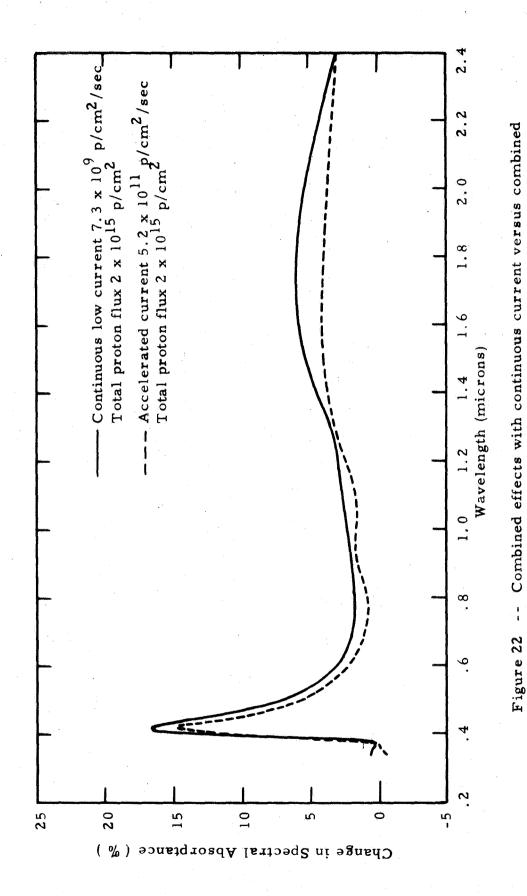


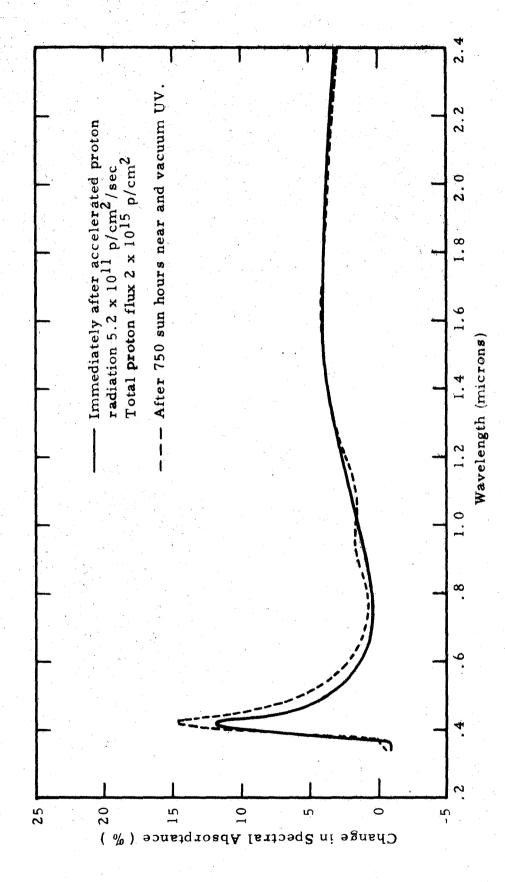
Figure 21 -- Combined effects versus sum of individual effects. Accelerated current - ZnO/K2SiO₃ (F-1-47-D)



ZnO/K2SiO3 (F-1-47-D)

effects with accelerated current.

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Effect of combined environment immediately after accelerated proton exposure and at the end of the test - ZnO/K_2SiO_3 (F-1-47D) Figure 23 --

sample and the algebraic sum of the individual irradiations on the other two samples for two separate runs of high and low proton flux. Figure 22 shows a comparison between the combined environment of vacuum, ultraviolet and protons of low and high flux rate. Figure 23 shows the effect of ultraviolet radiation and vacuum on a sample irradiated with protons at the beginning of a test and comparing the change in spectral absorptance from the irradiation measurement to the end of the test.

b. ZnO/Silicone

1. Protons

The effects of proton radiation are shown in Figures 24 and 25. The characteristic curve for ZnO susceptibility to proton damage is depicted. The effect of the accelerated protons is almost identical to that produced by the continuous low current indicating no rate effect. A slight difference is observed in the height of the change of absorptance which is greater for the continuous current. Also shown by Figure 24 is that the ZnO/Silicone showed a bleaching in the IR due to remaining in the vacuum chamber for approximately 74 hours. The effect of increasing the total proton flux to 1×10^{16} p/cm² is shown in Figure 25. peak change in absorptance was almost doubled with approximately 5% greater damage in the IR range.

2. Ultraviolet Effects

The effect of ultraviolet radiation on this material is shown in Figure 26. It shows very little damage with a slight absorptance peak near $.4\mu$ and less in the infrared than the $\rm ZnO/K_2SiO_3$. Both runs on the materials

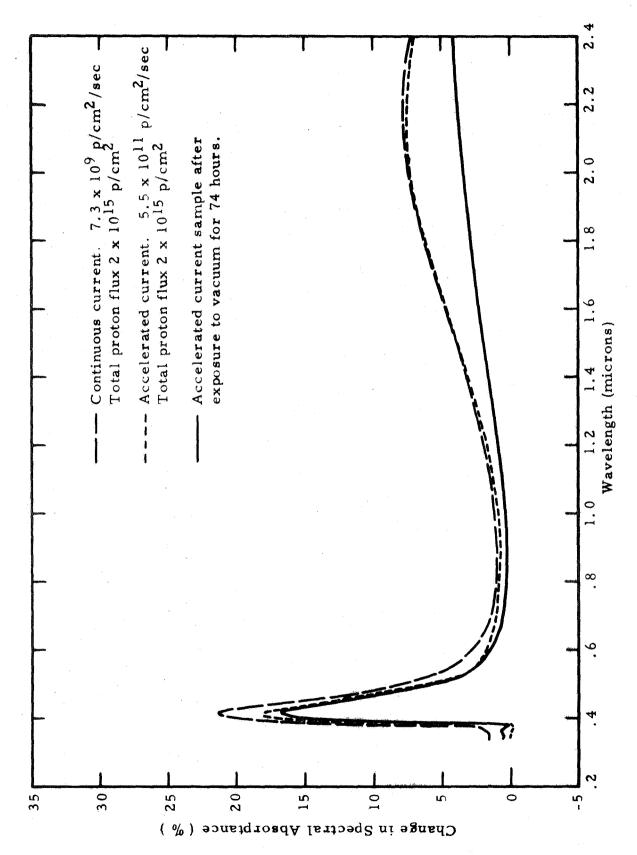
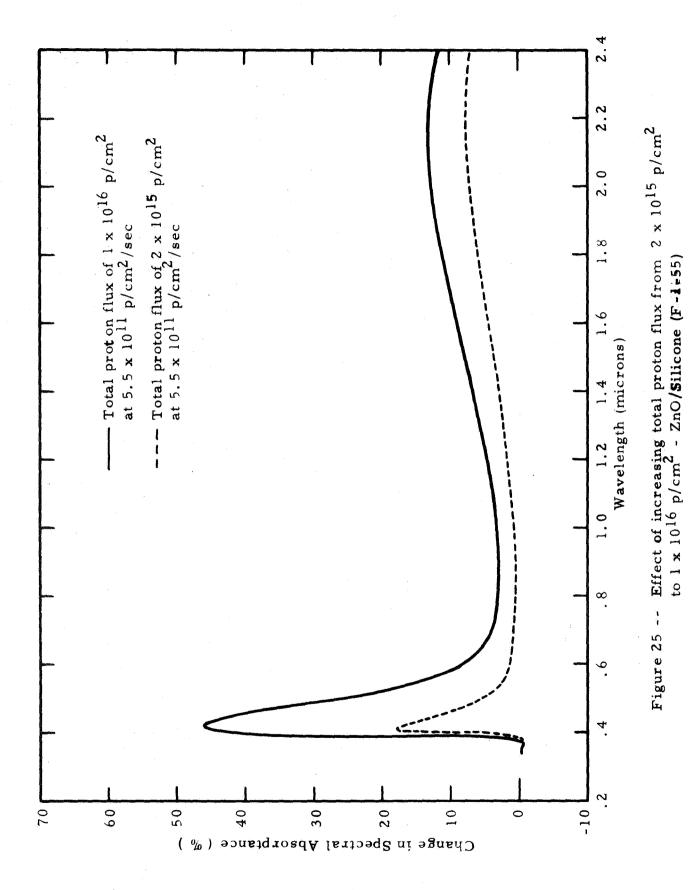
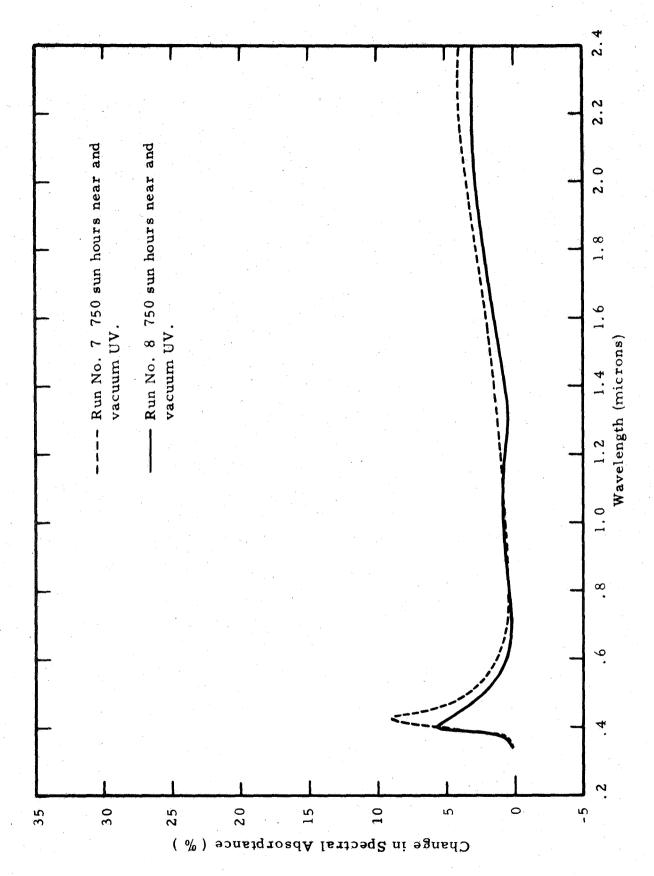


Figure 24 -- Rate and vacuum effect of proton radiation only - ZnO/Silicone (F-1-55)





Effect of UV radiation only. ZnO/Silicone (F-1-55) Figure 26 --

produced almost the same results as shown in this figure.

3. Combined Effects

The effects of the combined environment are shown in figures 27, 28, 29, and 30. Figures 27 and 28 show the comparison between the sum of the individual environment and the combined environments for both the high and low currents. Figure 29 shows that the absorptance peak around .4 μ was considerably greater for the continuous low current with approximately the same damage in the IR range. Figure 30 shows a greater change in spectral absorptance around .4 μ for the ultraviolet radiation than was effected in the ZnO/K₂SiO₃.

c. Lanthanum Oxide/Potassium Silicate (F-1-53)

1. Protons

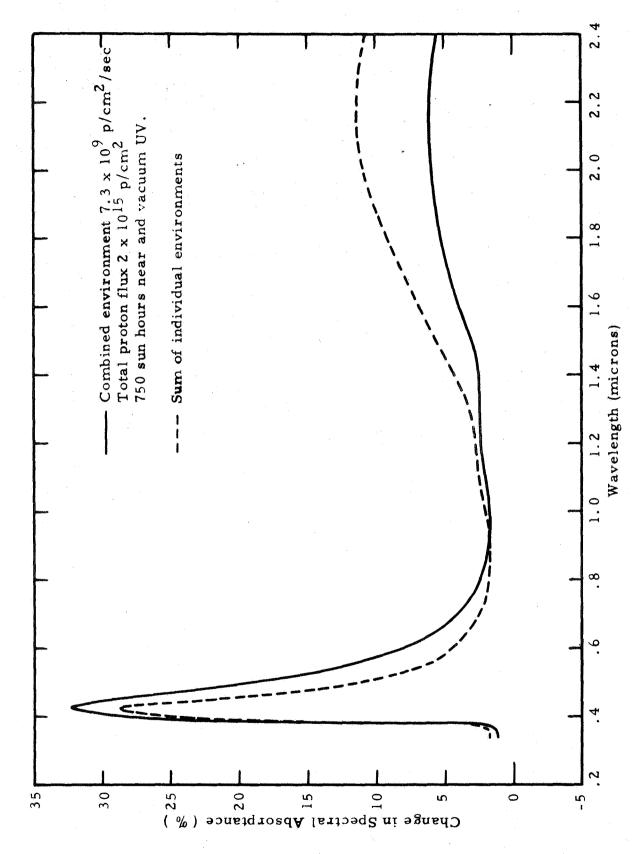
Figure 31 shows the effect of proton radiation on this material and the subsequent effect of exposure to a vacuum. Proton damage was very slight with practically no change due to vacuum exposure after irradiation.

2. Ultraviolet Effects

750 sun hours of ultraviolet and Lyman-alpha radiation produced the greatest damage to this material as shown by Figure 32. The effect was an absorptance peak in the near ultraviolet portion of the spectrum with a trailing off through the visible to practically no damage in the IR range.

3. Combined Effects

The effects of combined environment are shown in Figures 33 and 34. The algebraic



Combined effects versus sum of individual effects. Continuous low current - ZnO/Silicone (F-1-55) Figure 27 --

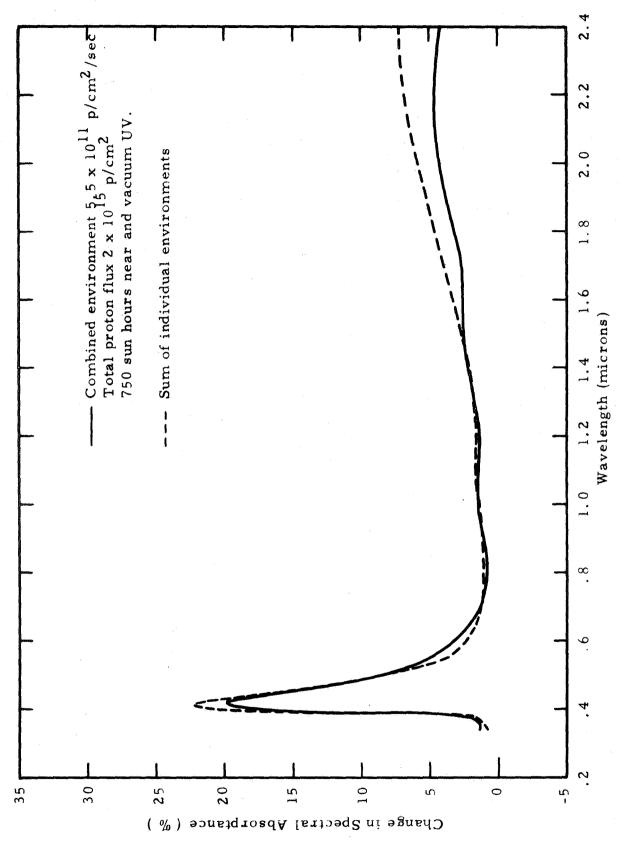
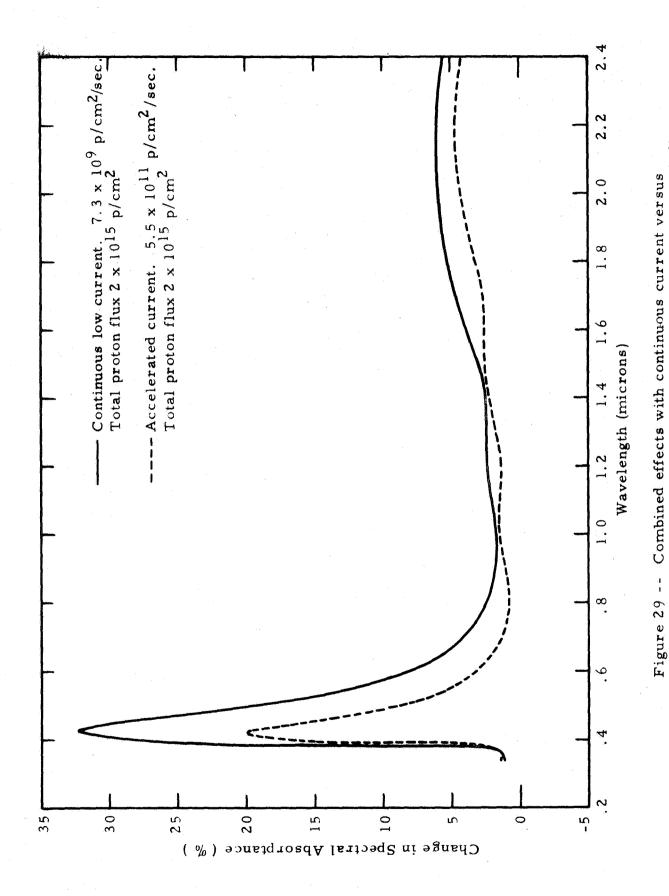


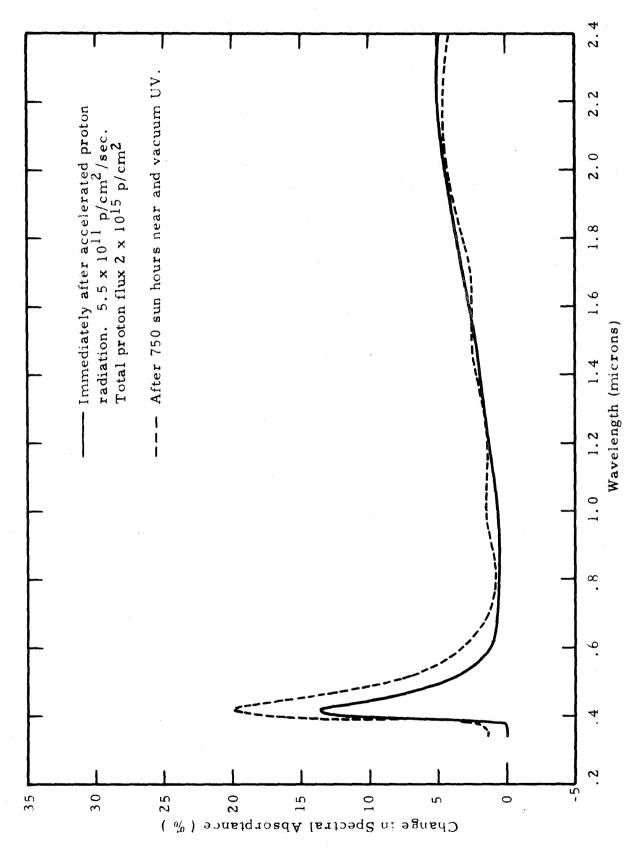
Figure 28 -- Combined effects versus sum of individual effects.

Accelerated current - ZnO/Silicone (F-1-55)

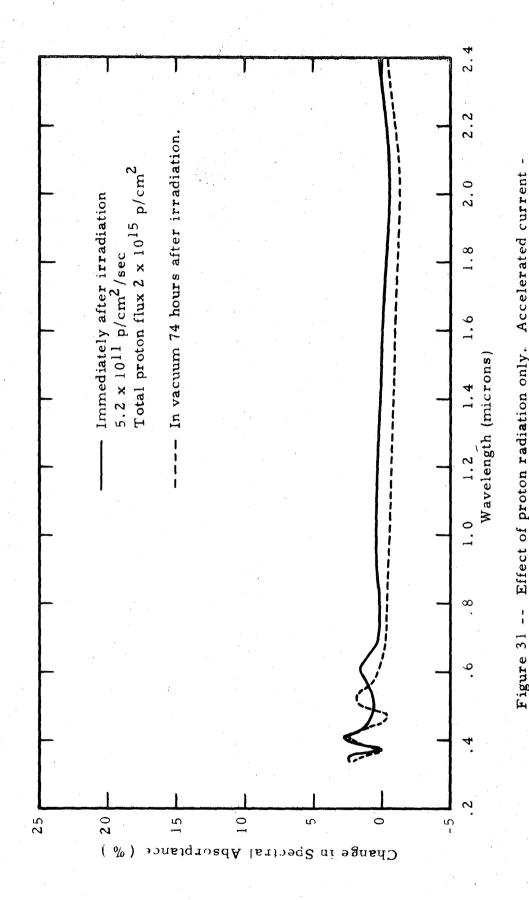


combined effects with accelerated current. ZnO/Silicone (F-1-55)

-53-



Effect of combined environment immediately after accelerated proton exposure and at end of test. ZnO/Silicone (F-1-55) Figure 30 --



immediately after irradiation and at end of test

Figure 31 --

 $\text{La}_2 O_3/\text{K}_2 \text{SiO}_3 \text{ (F-1-53A)}$

- 5**5** -

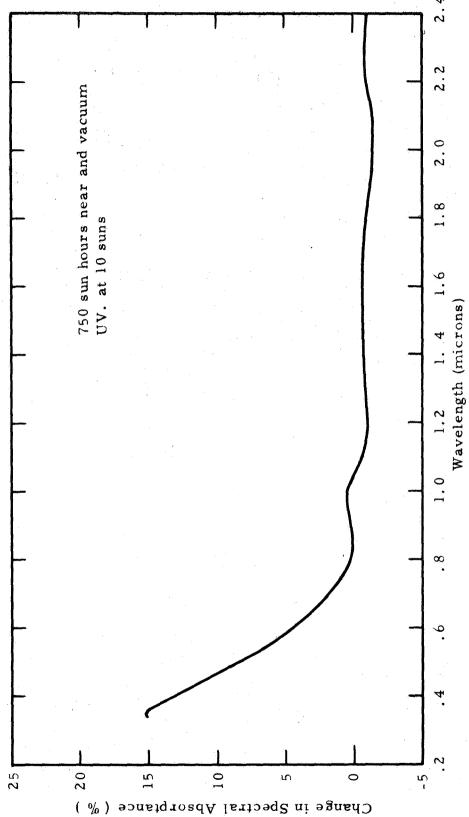
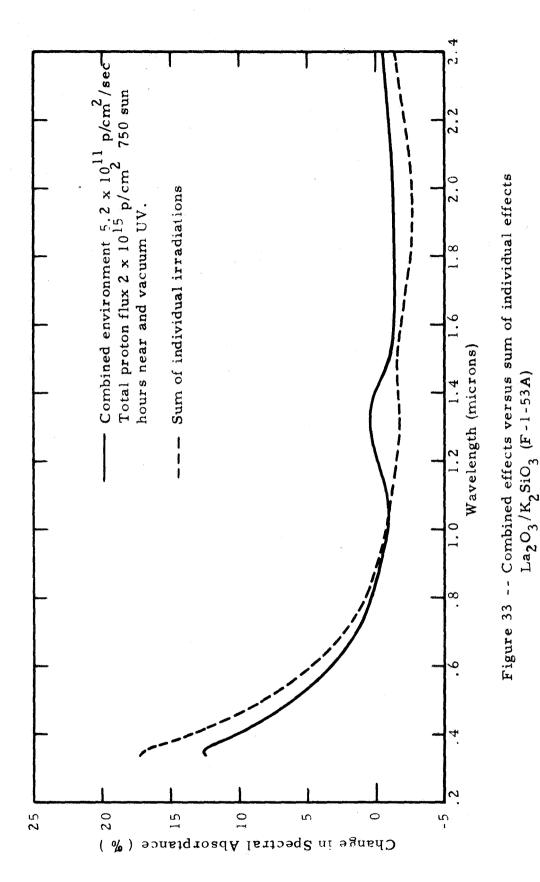
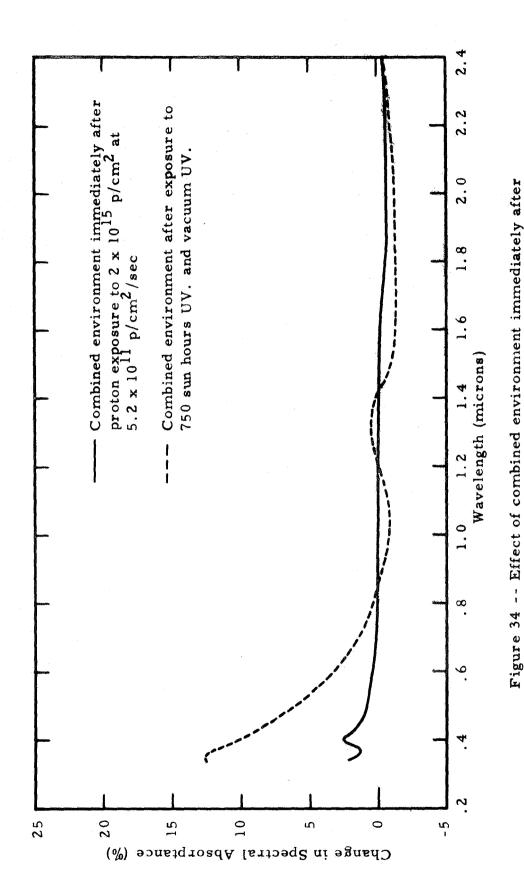


Figure 32 -- Effect of UV radiation only. La2O3/K2SiO3 (F-1-53A)





accelerated proton exposure and at end of test

La2O3/K2SiO3 (F-1-53A)

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sum of the individual damage very nearly equalled the combined environment damage. Figure 34 shows that practically all of the damage to the material in the combined environment was due to the solar electromagnetic radiation.

d. Lanthanum Oxide/Potassium Silicate (F-1-38)

1. Protons

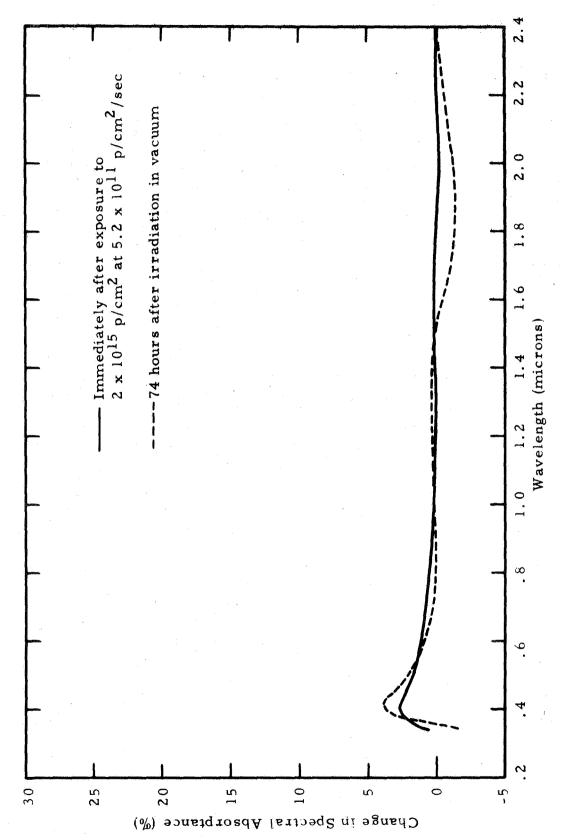
The effect due to proton radiation only is shown in Figures 35 and 36. This was a different batch of Lanthanum Oxide/
Potassium Silicate, however, it showed practically the same effect due to protons as did the F-1-53 material. Increasing the total proton flux by five times produced very little more damage as shown by Figure 36.

2. Ultraviolet Effect

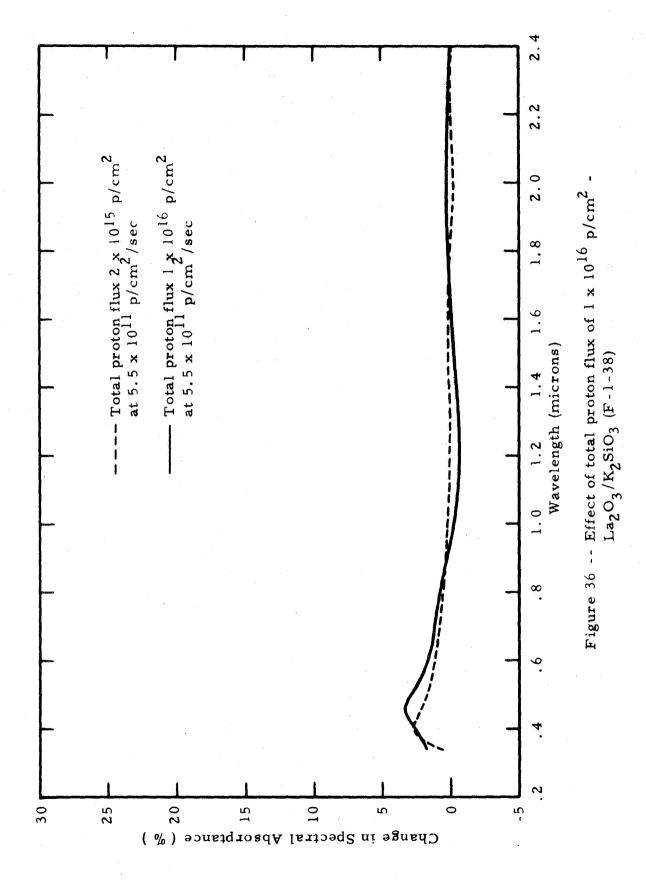
The absorptance peak produced by the electromagnetic radiation was considerably greater in this batch of Lanthanum Oxide (F-1-38) than in that reported in the previous paragraph (F-1-53). This is shown in Figure 37. Damage in the IR range was again practically zero as before.

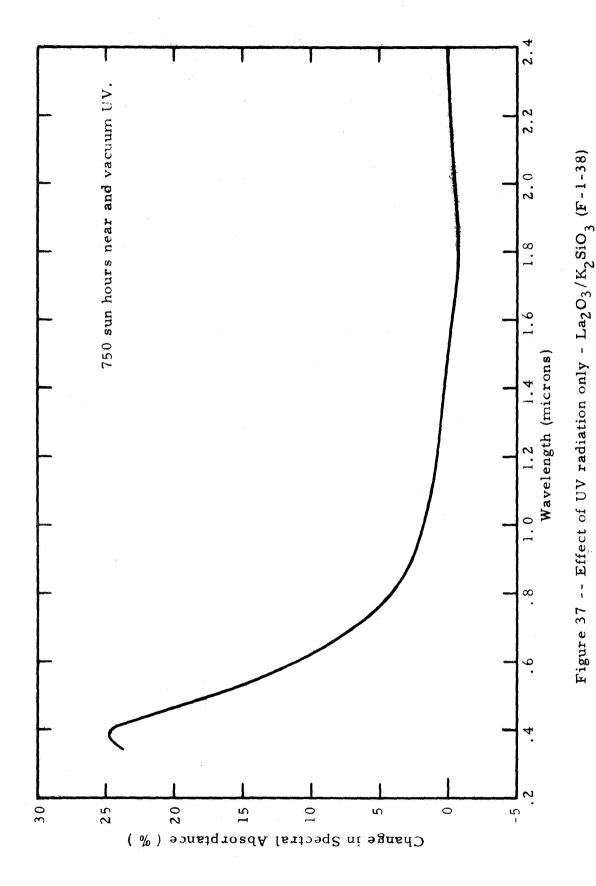
3. Combined Environment

The effects of combined environment are shown in Figures 38 and 39. The principal damage is seen to be due to the electromagnetic radiation with the algebraic sum very nearly equalling the combined damage.



immediately after radiation and at end of test $\text{La}_2\text{O}_3/\text{K}_2\text{SiO}_3$ (F-1-38) Figure 35 -- Effect of proton radiation only - Accelerated current -





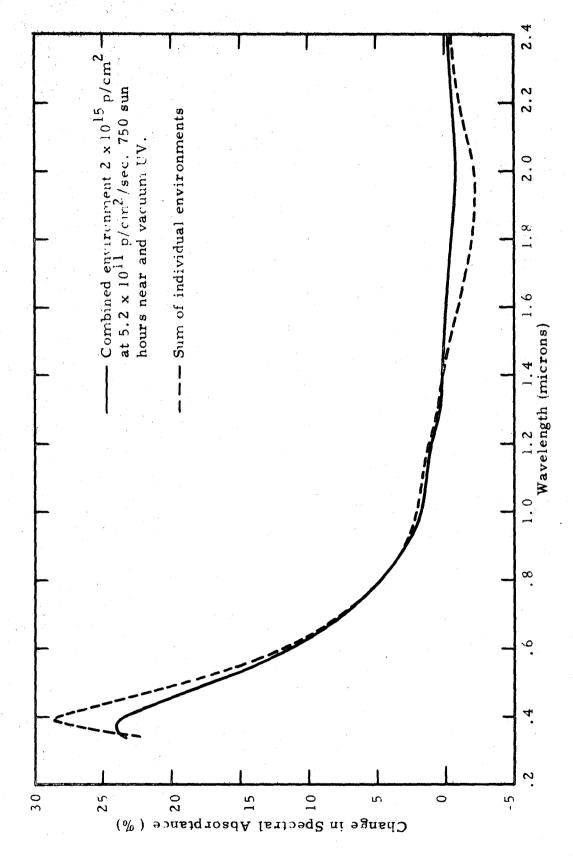
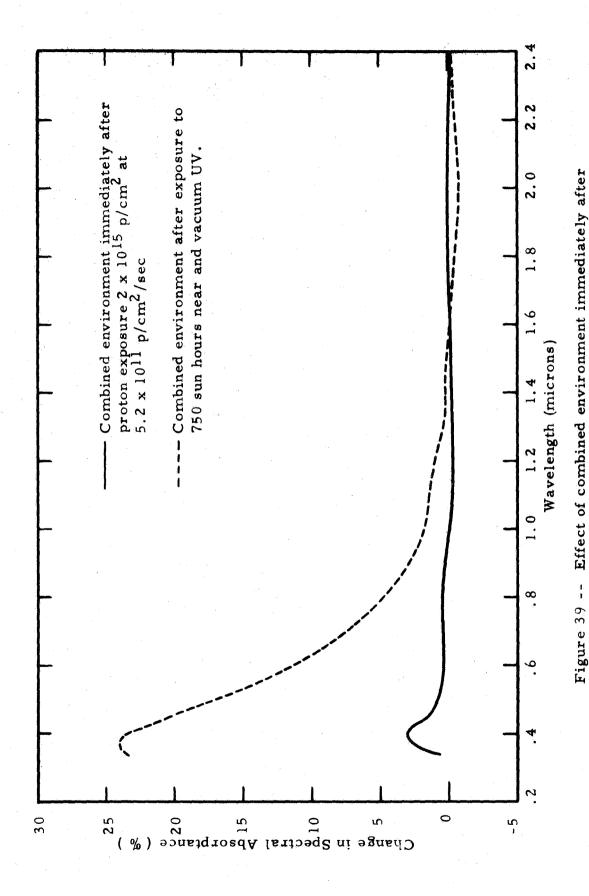


Figure 38 -- Combined effects versus sum of individual effects -La2O3/K2SiO3 (F-1-38)



accelerated proton exposure and at end of test -

La2O3/K2SiO3 (F-1-38)

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e. **S**-13**G**

1. Proton Effects

The effect of proton radiation on this material is shown in Figure 40. The material showed the characteristic damage curve for ZnO with about the same effects as the ZnO/Silicone irradiated with continuous low current.

2. Ultraviolet Effects

The effect of ultraviolet radiation only is shown in Figure 41. The change in solar absorptance is greater around .4 micron than the ZnO/Silicone or the ZnO/K₂SiO₃ with virtually no damage again in the IR range.

3. Combined Environment Effect

The effect of combined environment simulation is shown in Figure 42. Bleaching of the proton damage in the IR range has apparently occurred.

f. TiO2 Silicone

These tests were conducted in order to compare the results of in situ testing of TiO₂ Silicone with the work of Pinson⁽²⁾ which was conducted non-in situ.

1. Proton Effects

Figure 43 shows the effect of proton radiation on TiO₂/Silicone. It has the characteristic peak of ZnO but does not return to near zero in the visible range as does the ZnO. This material received 1-1/2 times more total proton flux than did the other materials.

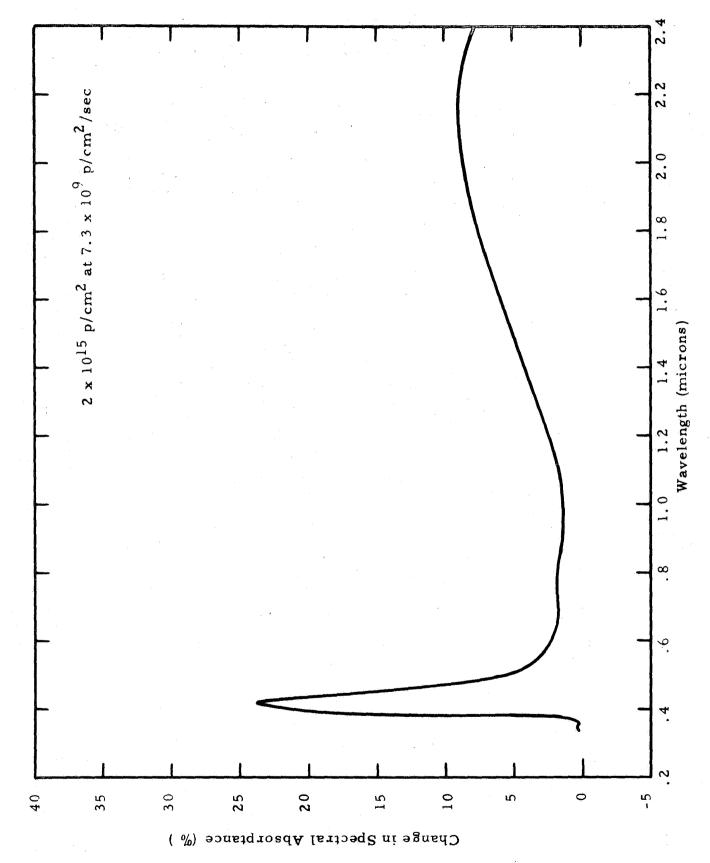
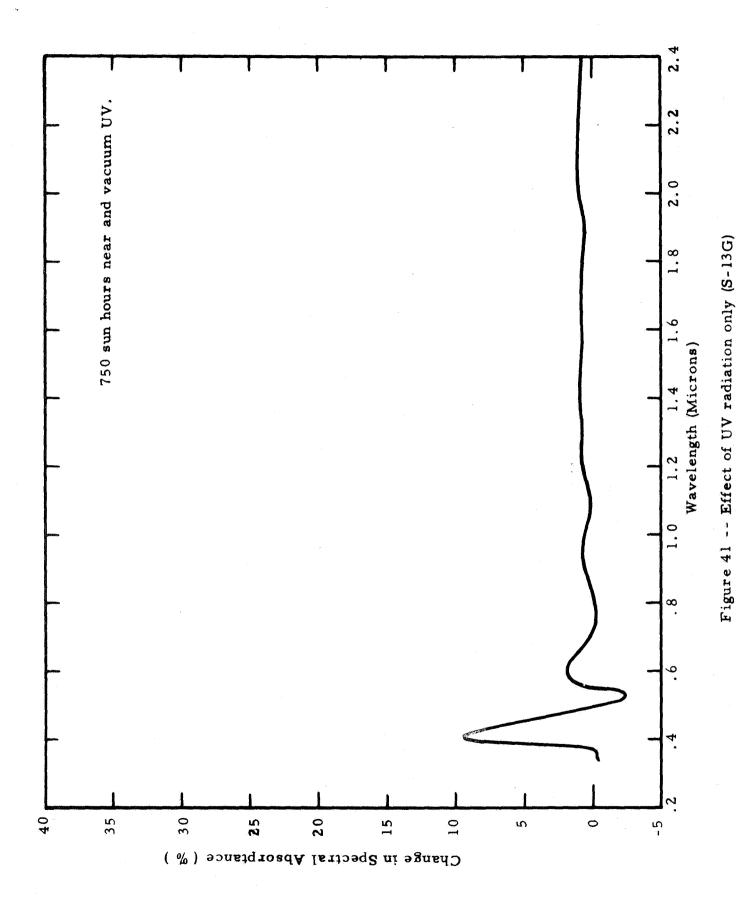


Figure 40 -- Effect of proton radiation only (S-13G)



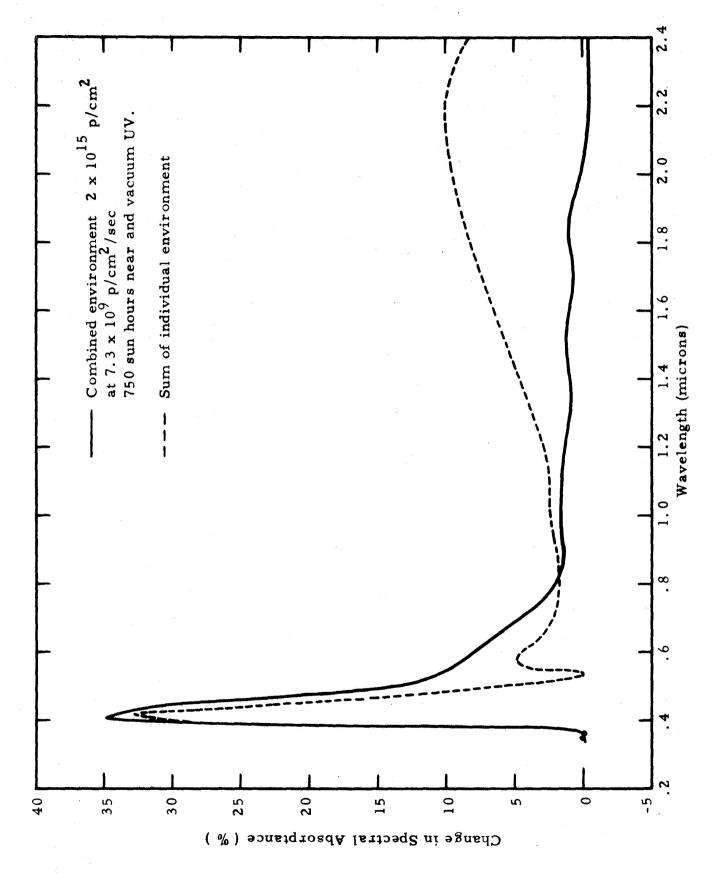


Figure 42 -- Combined effect versus sum of individual effects (S-13G)

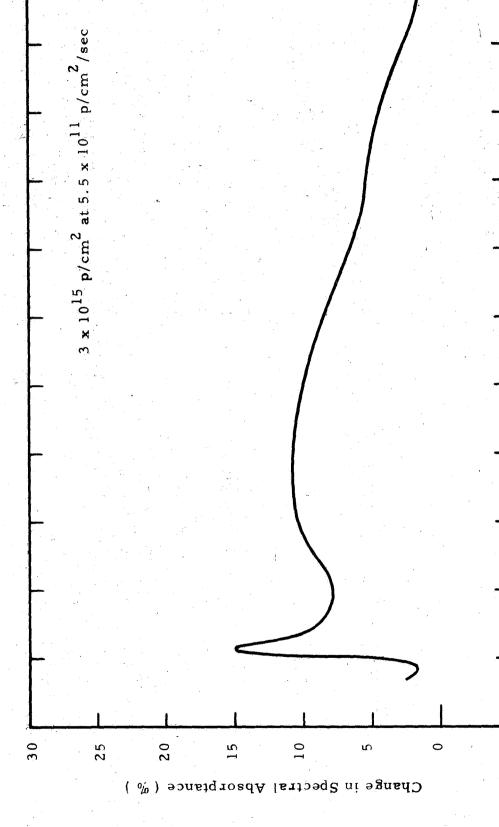


Figure 43 -- Effect of proton radiation only (TiO2 Silicone)

1.2 1.4 Wavelength (microns)

 ∞

2. Ultraviolet Radiation Effect

As shown by Figure 44, practically no damage was evident due to the near ultraviolet and vacuum ultraviolet radiation. This sample, however only received a total of 190 sun hours of electromagnetic radiations.

3. Combined Environment Effect

The effect of combined environment is shown in Figure 45. It may be seen that the combined damage is less than the proton damage alone which is just the opposite of the results obtained by Pinson⁽²⁾ Synergistic effects were noted in Pinson's tests in which the combined damage was greater that the sum of the individual environments.

g. Powdered Samples

Powdered samples of SiO_2 , La_2O_3 , TiO_2 , Al_2O_3 and MgO were all irradiated with protons only for a total flux of 5×10^{15} p/cm² at a rate of 5.5×10^{11} p/cm²/sec. The effects are shown in Figures 46 through 50. The only powdered sample which had significant damage is the TiO_2 . As may be seen from these figures, the other samples exhibited little or no damage.

B. Non-In Situ Tests

Three exposures were conducted <u>non-in situ</u> using a 5 KW mercury xenon lamp in the solar simulator. The results of these tests are discussed in the following paragraphs based upon the environmental parameters selected. The curves which accompany show only changes in spectral absorptance.

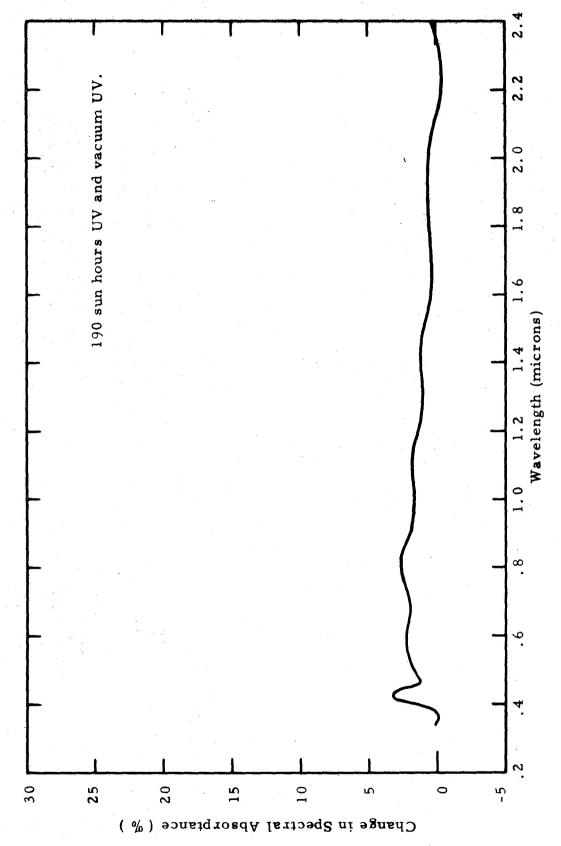


Figure 44 -- Effect of UV radiation only (TiO2 Silicone)

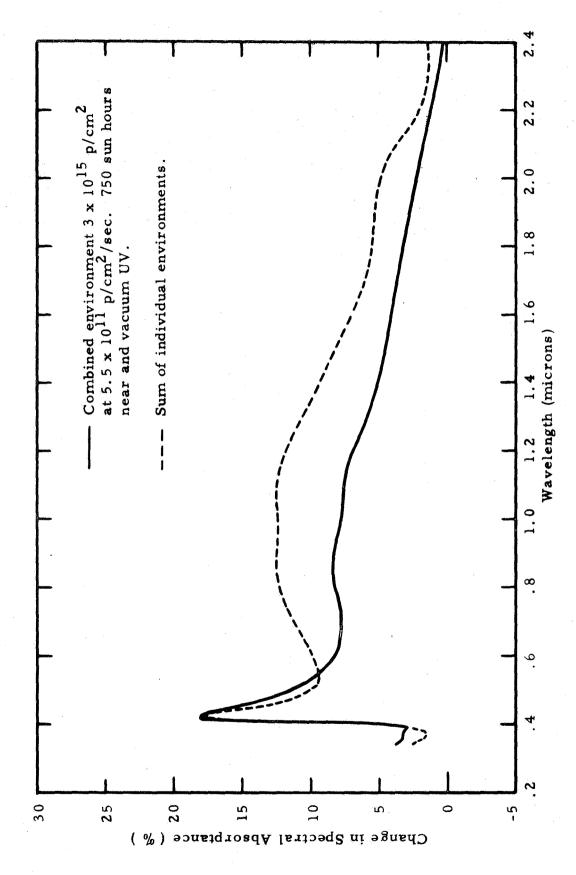


Figure 45 -- Combined effects versus sum of individual effects (TiO2 Silicone)

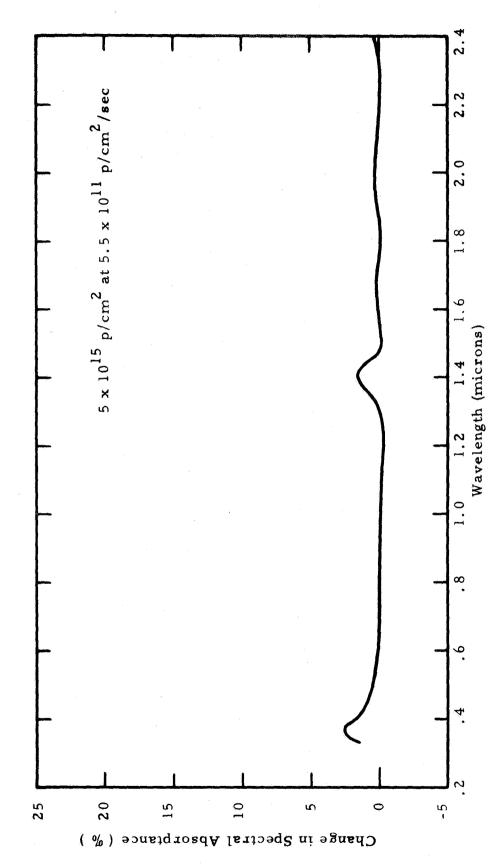


Figure 46 -- Effect of proton radiation only (SiO₂ powdered)

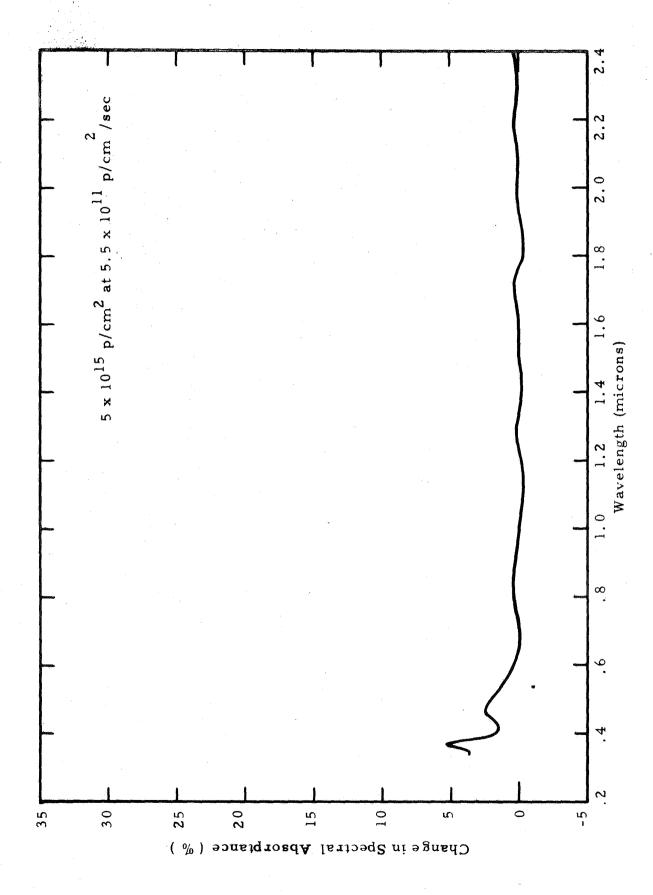


Figure 47 -- Effect of proton radiation only (La2O3 powdered)

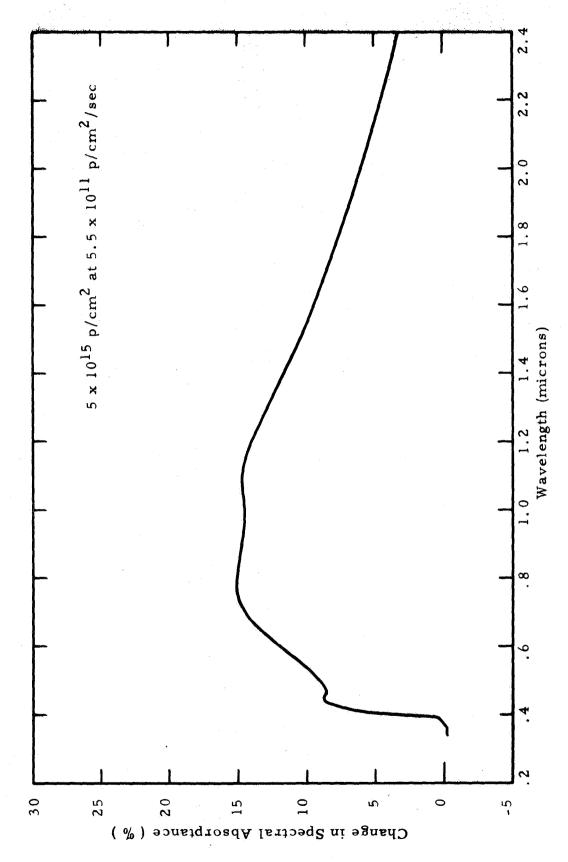


Figure 48 -- Effect of proton radiation only (${\rm TiO_2}$ powdered)

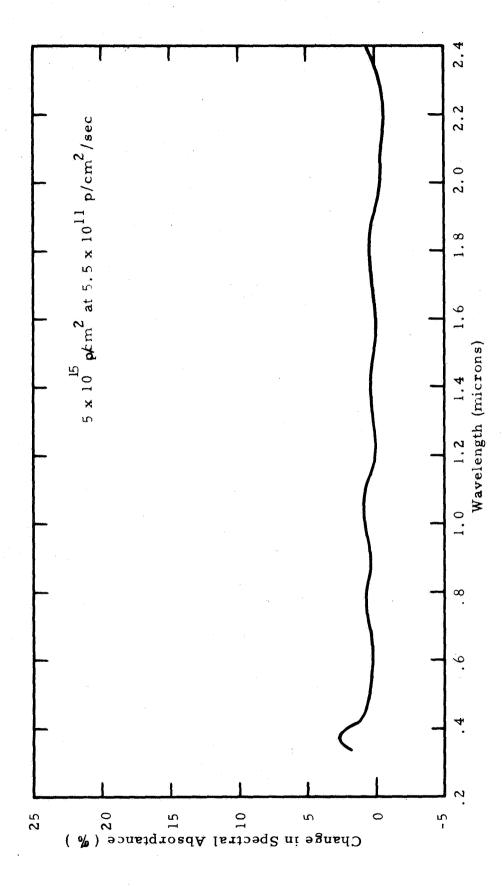
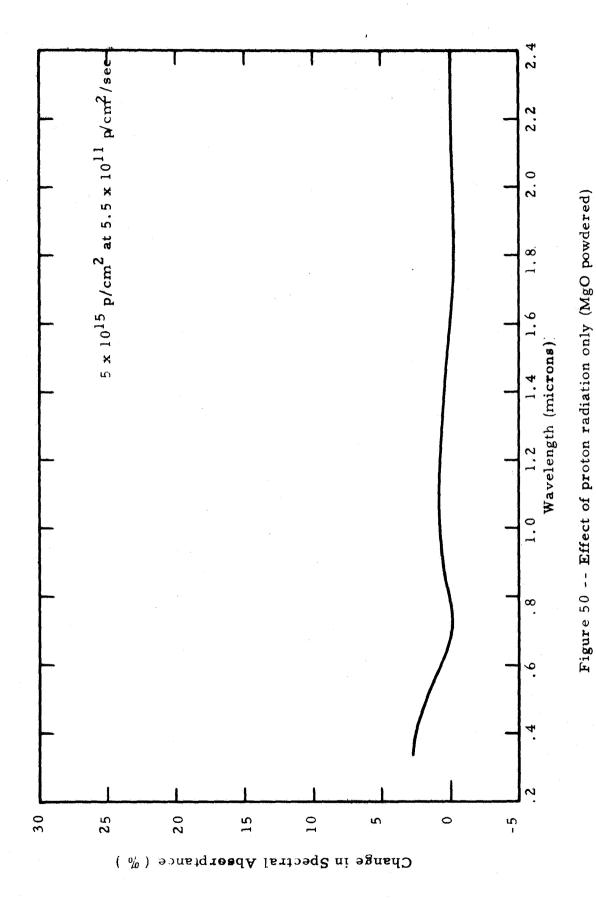


Figure 49 -- Effect of proton radiation only (AL_2O_3 powdered)



-7**7**-

1. SP 500 Zinc Oxide

Figure 51 shows the change in spectral absorptance for compressed zinc oxide powder following exposure to 10 Kev protons. Total flux was 2×10^{15} protons/cm² at 5.5×10^{11} p/cm²/sec. Sample temperature was 298° K. Damage in this case was comparable to that for ZnO/K₂SiO₃ in the same environment, from earlier work, indicating that the damage for the latter is occurring in the pigment.

Figure 52 shows the effect of a combined proton and ultraviolet environment on zinc oxide. The environment included a total 10 Kev proton flux of 2×10^{15} p/cm² at 7.4×10^9 p/cm²/sec and 750 sun hours of near and vacuum UV at 10 solar equivalents. The damage was substantially less than for the sample exposed to protons only, indicating proton damage bleaching by electromagnetic radiation.

Figure 53 shows the effect of exposure to 750 sun hours of near and vacuum UV at 10 solar equivalents. Measured damage was very small and essentially within the error of readings. Bleaching of damage undoubtedly occurred upon return of the sample to atmospheric conditions.

2. Vacuum Deposited Ag on Fused Silica

Figure 54 shows the effect of exposure to combined 10 Kev protons (1.5 x 10^{17} p/cm² at 5.5 x 10^{11} p/cm²/sec) and 750 sun hours of near and vacuum UV on vacuum deposited silver on fused silica. Substantial damage incurred apparently from the proton exposure. The peak in the induced absorption band occurred at about 0.365 micron.

Figure 55 demonstrates the effect of 750 sun hours of near and vacuum UV at 10 solar equivalents to the vacuum deposited silver on fused silica. Changes were essentially within the errors in measurements.

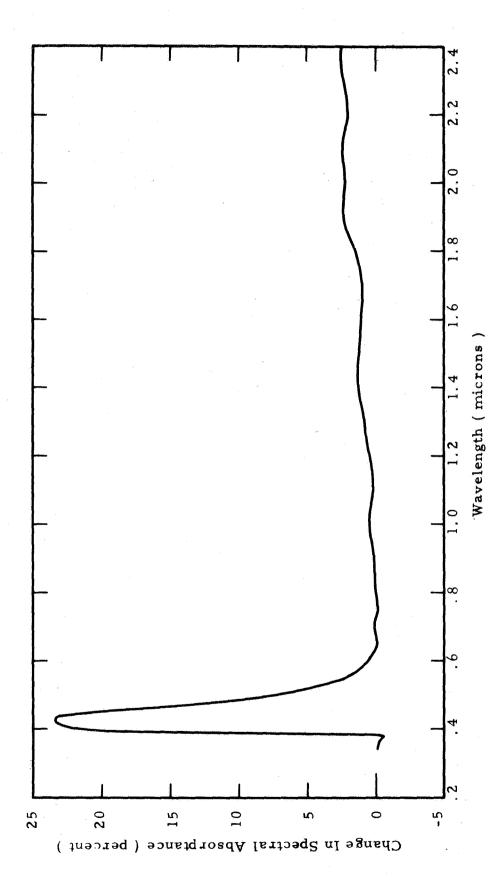


Figure 51--Effect of proton exposure on SP 500 zinc oxide at 298°K. 10 Kev protons - 2 x 10¹⁵ p/cm² at 5.5 x 10¹¹ p/cm²/sec.

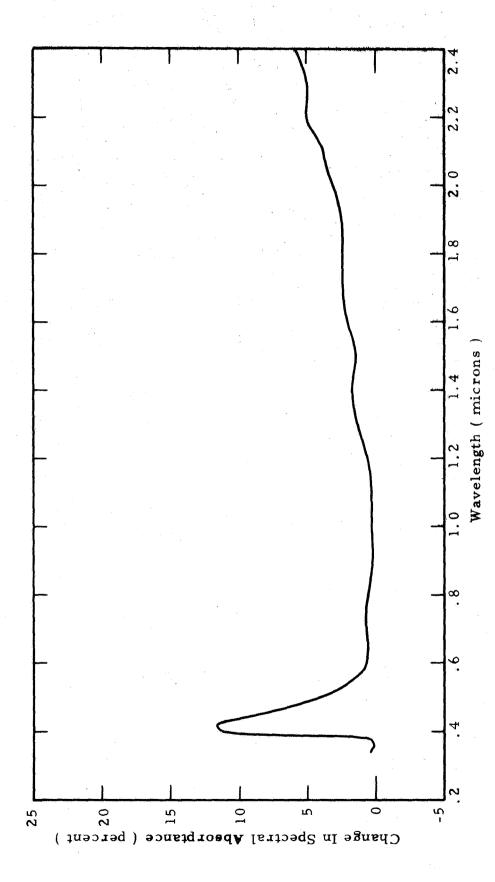


Figure 52--Effect of combined environment exposure on SP 500 zinc oxide at 2990K. 10 Kev protons - 2 x 10¹⁵ p/cm² at 7.4 x 10⁹ p/cm²/sec. 750 sun hours of near and vacuum UV at 10 solar equivalents.

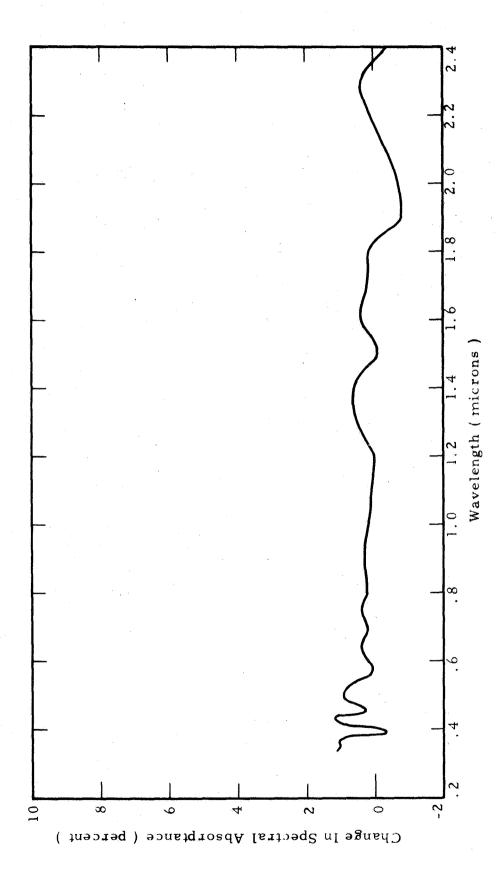
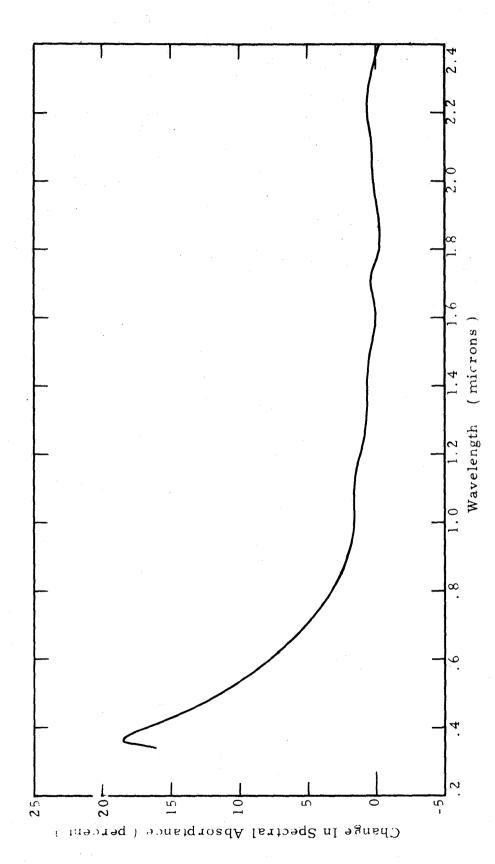


Figure 53--Effect of ultraviolet exposure of SP 500 zinc oxide at 299°K. 750 sun hours of near and vacuum UV at 10 solar equivalents.



deposited Ag on fused silica at $304^{\circ}K$. 10 Kev protons -1.5 x 10^{17} p/cm² at 5.5 x 10^{11} p/cm²/sec. 750 sun hours of near and vacuum UV at 10 solar Figure 54-- Effect of combined environment exposure on vacuum equivalents.

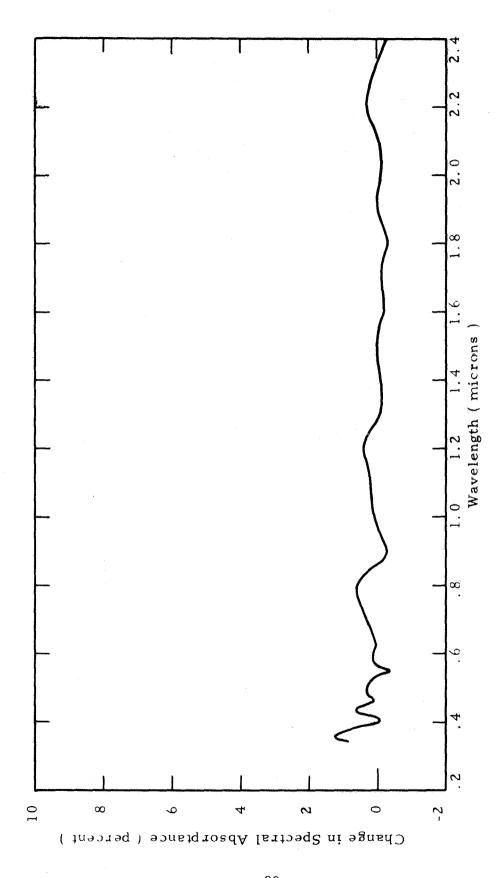


Figure 55--Effect of ultraviolet exposure on vacuum deposited Ag on fused silica at 3040K. 750 sun hours of near and vacuum UV at 10 solar equivalents.

VII. CONCLUSIONS AND RECOMMENDATIONS

A. Conclusions

- 1. The ZnO/potassium silicate is more stable in an ultraviolet radiation environment than in a proton environment. It is apparently very susceptible to proton damage in the spectral range studied. A threshold of approximately 10¹⁵ p/cm² causes measurable damage with proportionate damages for doses in excess of this level.
- 2. There was not evidence to indicate that synergistic effects existed for the ZnO/K₂SiO₃ material. In all but one test, the combined environment tests including both protons and ultraviolet radiation produced less optical damage than the sum of the damages due to the individual environments.
- 3. There is definite evidence that the rate at which protons are applied to ZnO/K_2SiO_3 has a definite effect on the amount of damage to the material, especially in the infrared portion of the spectrum.
- 4. The 1-11-9-13 ZnO/K₂SiO₃ demonstrated comparable degradation effects to the F-1-47-D ZnO/K₂SiO₃. The 1-11-9-13 did exhibit less damage in the IR portion of the spectrum but had a larger change in absorptance near .4 micron.
- 5. The Lanthanum Oxide/Potassium Silicate is more susceptible to ultraviolet radiation damage and less susceptible to proton damage. Increasing the total protons by a factor of 5 did not increase the damage indicating a very good resistance to protons. In contrast to the ZnO/K₂SiO₃, the La₂O₃/K₂SiO₃ shows a definite damage effect, principally due to ultraviolet exposure. Of the two batches of La₂O/K₂SiO₃ tested, the F-1-38 was the least resistant to ultraviolet damage.

- 6. Combined environment tests including both protons and ultraviolet radiation produced comparable damage to the sum of the individual environments for the Lanthanum Oxide/Potassium Silicate.
- 7. The TiO₂/Silicone is highly susceptible to proton damage and has very little susceptibility to ultraviolet radiation. This effect also showed up in the powdered samples since the TiO₂ was the only powder which showed significant damage due to proton radiation.

B. Recommendations

- 1. Further study should be conducted on the influence of the electromagnetic spectral distribution on the combined environment synergistic effects.
- 2. The reciprocity effects of accelerated testing for both electromagnetic and particulate radiation should be investigated.
- 3. Temperature effects should be investigated for materials studied in situ.
- 4. The effects of simulated space electron radiation should be studied.
- 5. The coatings should be subjected to proton and electron particle irradiation in order to establish the relation between particle energy and optical degradation.

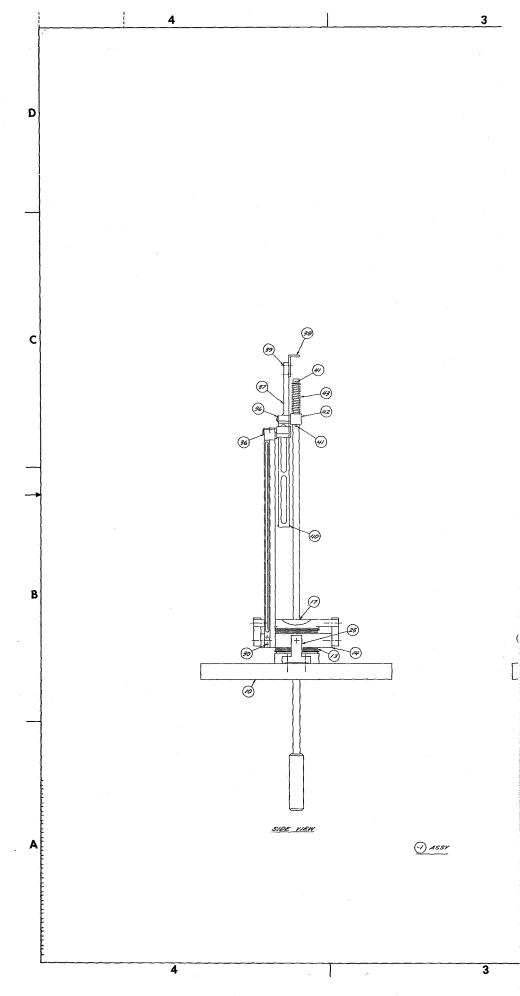
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IX. APPENDIX

Following are prints of dimensional information for items fabricated under this contract.



EC SYM ZONE DATE APPROVE D C 6. PLACE -14 GIMBAL RING OVER ITEM 13 BELLOWS BEFORE MAKING FINAL WELD 3> ALLIED BEARING SUPPLY CO., BZZ SO. BOULDER AVE, TULSA, OKLA. METAL BELLOWS CORP., 20977 KNAPP ST., CHATSWORTH, CALIF. 334(35) 3> .625 MAX O D , .453 MIN I D , 3.00 FREE LENGTH , 1.25 MAX SOLID HEIGHT , 541 LEJIN SPRING RATE 2> 2024-7351 AL ALY 1> TYPE 304 STS 27 MAKE FROM V4-ZOUME XI LG BUTTON NO CAPSON COMPRESSION SPANNS @ FINISHED BNDS PIVOT - SLIDE 4 -44 575 1 COME 3 575 43 -42 2> 42 2 1/8 DIA X 3/4 LG SPRING PIN 575 41 SPRING PIN PAN HEAD SCREW 1 1/16 DIA X 7/8 LG 575 40 2 #4-40 UNC X % LG 575 39 1 -38 FORK 38 PIVOT ARM

"INDUSTRIAL"

RETAINING RING

82° FLAT HEAD

SCREW / -37 37 2 1000-25 575 36 4 #4-40 UNC X 3/6 LG STS 35 24 LIACUUM TIGHT FUSION CAP. (2) 1 -34 2> 34 SLIDE-PIVOT OYAL POINT SETSCREW 2> 33 -33 575 2 48-32 UNC 1/4 LG 32 2 -3/ 31 BUTTON NEAD CAP SCREW 4 410-24 UNC X 1 LG 575 30 -29 CROSSPIECE 29 CANTILEVER ARM PIVOT ROD SUPPORT GIMBAL RING SUPPORT 2 28 -28 -27 SUBASSY 27 -26 26 SUPPORT

GIMBAL RING PIVOT

BUTTON HEAP

CAP SCREW

HARDENED & PRECISION

GROUND DOWEL PON

VOKE SUPPORT -25 SUBASSY 25 2 14-20 UNC X 1 LG 24 14DIAX 1 LG 23 572 -22 22 FOLD -ZI SUBASSY YOKE PIVOT 21 -20 HANDLE 20 7 ROD 19 -19 YOKE \triangleright -18 OPERATING ARM
TORRINGTON
NEEDLE BEARING
RING 1 -IT SUBASSY 8-47 16 -15 15 GIMBAL RING 1 -14 SUBASSY 14 TOP VIEW 1 50511 BELLOWS 13 12 -/2 NECK 11 A FLANGE 1 -10 SUBASSY PORT COVER

NOMENCLATURE OR
DESCRIPTION 10 OFF OTY PART OR ASSOCIATION OF ASSOCIATION OF ASSOCIATION OF ASSY NO. ZONE ITEM MATERIAL OR NOTE PART OR IDENTIFYING NO. LIST OF MATERIALS OR PARTS LIST - MAST NO.

- UNIESS OTHERWISE SPECIFIED.

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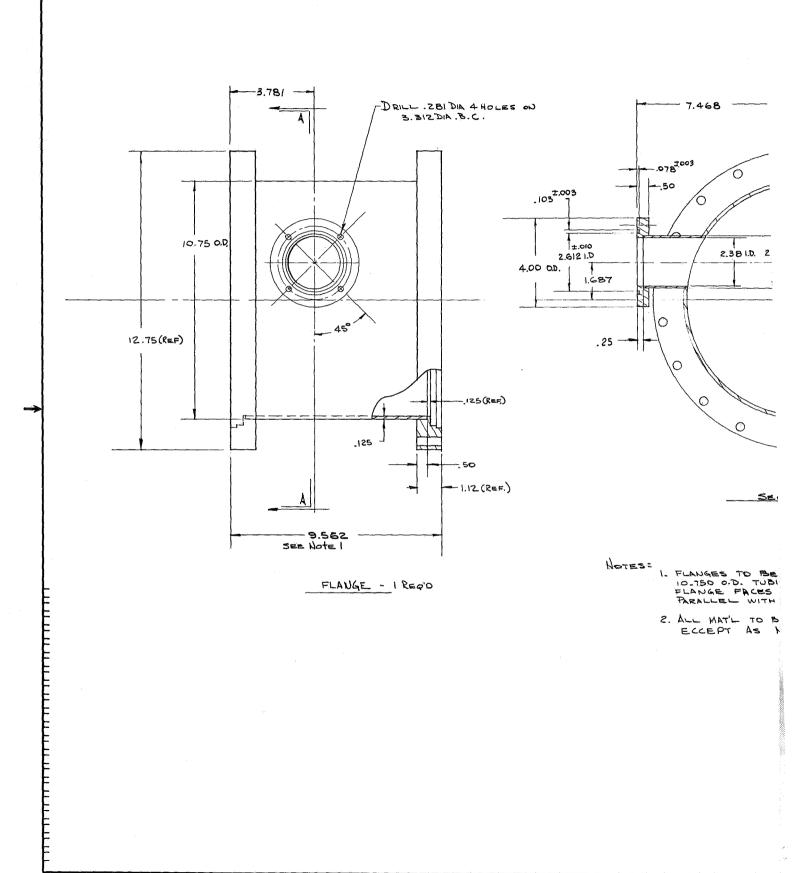
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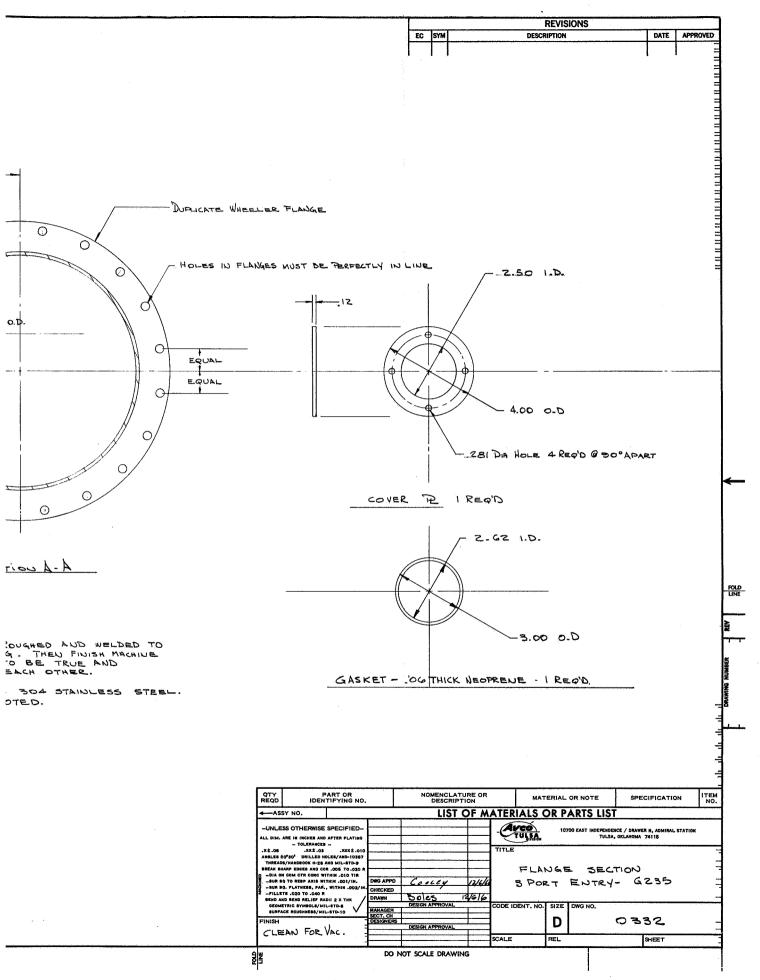
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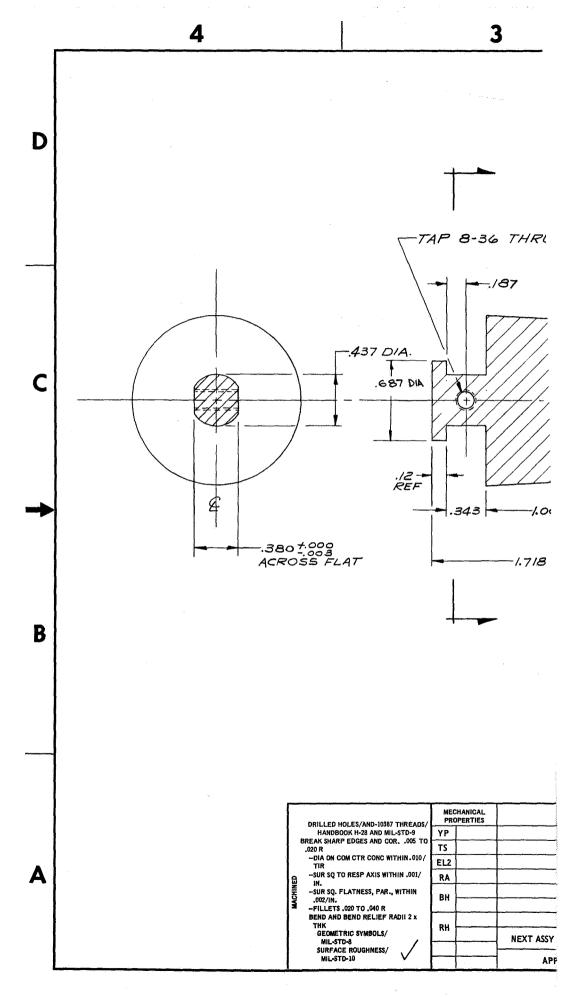
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Tules, Oklahoma TRANSFER MECHANISM-IN SITU REFLECTOMETER SUBTACE NO E SKT-0303 WT DASH NO. NEXT ASSY USED ON NEXT ASSY SER APPLICATION QTY REQD EFFE SHEET 1 OF 2 DO NOT SCALE DRAWING 2 1

2



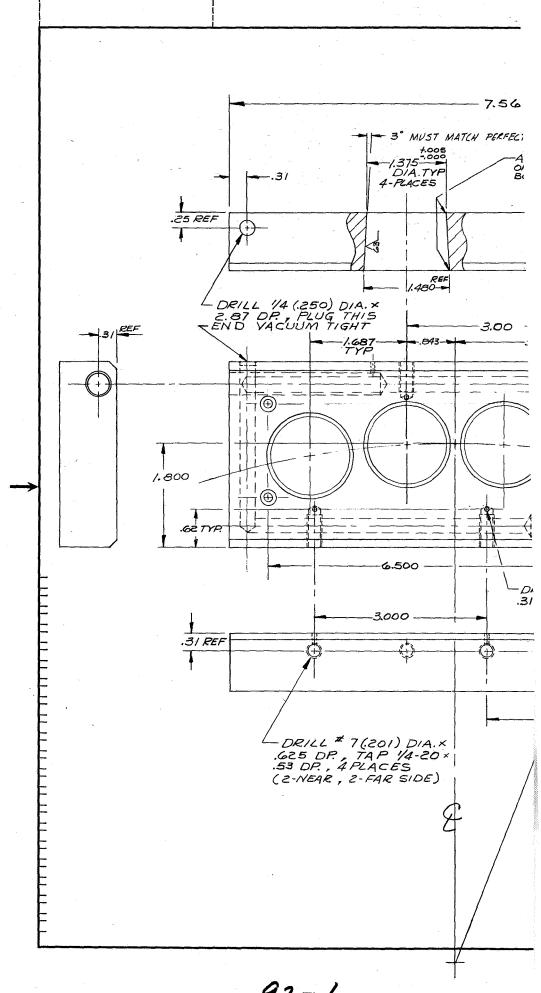


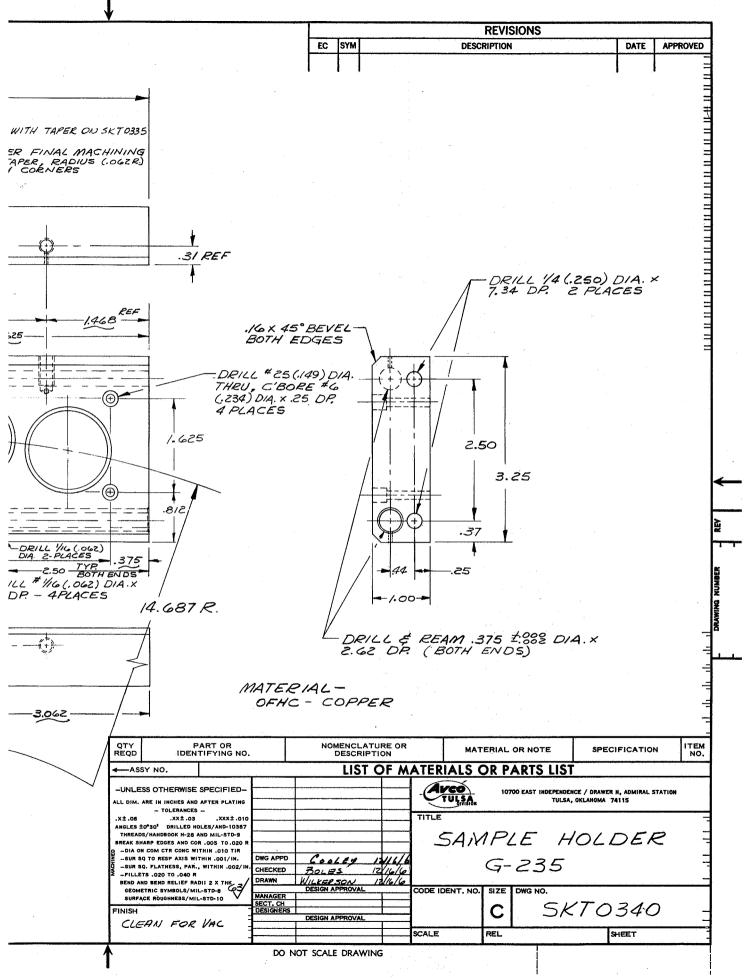


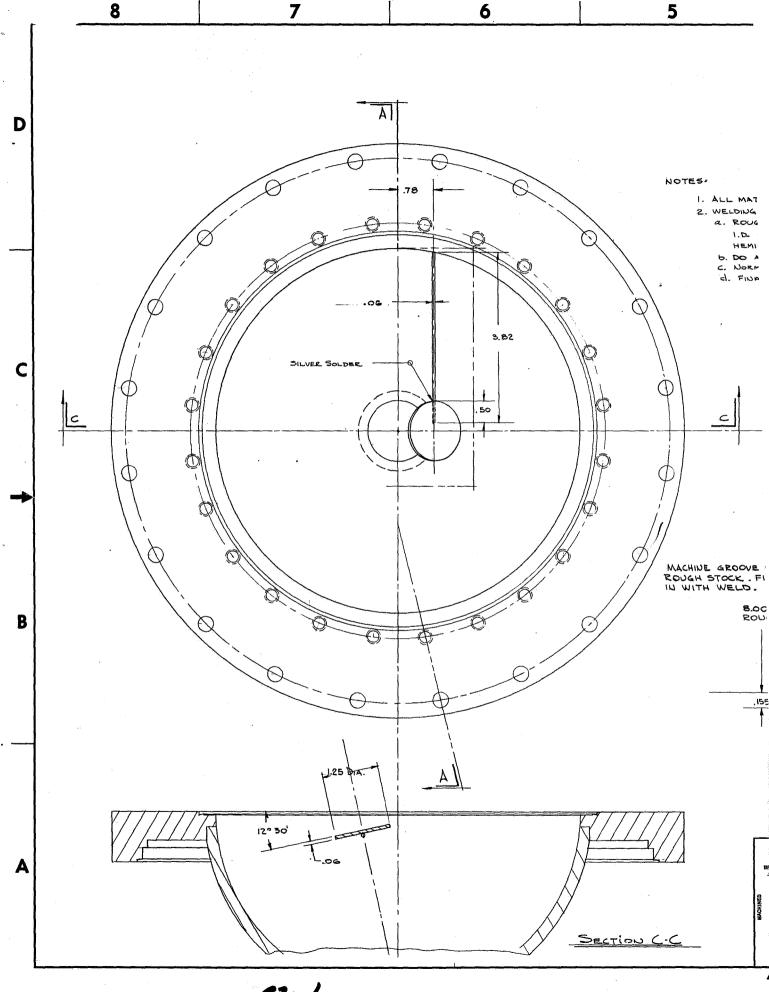
REVISIONS DESCRIPTION D MIN. UNDERCUT 32 THREADS/INCH SEE MATING PART SKT0336 TAPER ON SKT 0340 1.375 +000 DIA. +002 450. 1.158 1.162 MAJOR DIA. .080 .250 -.032 +003 SLOT -.0/5 MATERIAL - (OFHC) COPPER TOLERANCES .XX ±,030 .XXX ±,010 CLEAN FOR VACUUM NOMENCLATURE OR DESCRIPTION PART OR IDENTIFYING NUMBER MATERIAL OR NOTE SPECIFICATION REQD LIST OF MATERIALS OR PARTS LIST ASSY NO. UNLESS OTHERWISE SPECIFIED DIMENSIONS ARE IN INCHES TOLERANCES CONTRACT NO. AVCO CORPORATION TULBA DIVISION TULSA, OKLAHOMA FRACT DEC ANGLES DWG. NO. TITLE MATERIAL: SAMPLE PLUG COOLEY /2//6/6 DATE A G-235 BOLES 12/16/6 DRN WILKERSON CODE IDENT NO. DWG. NO. DESIGN APPROVED SIZE *5KT0335* USED ON C DESIGN APPROVED ICATION SCALE WEIGHT SHEET

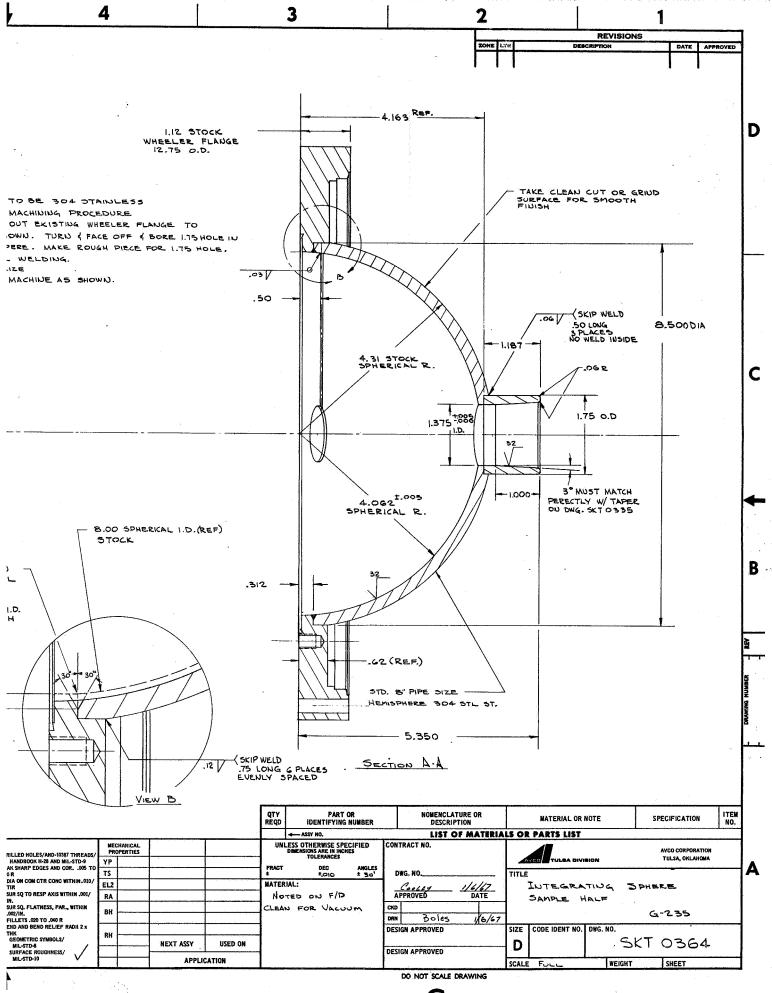
92-2

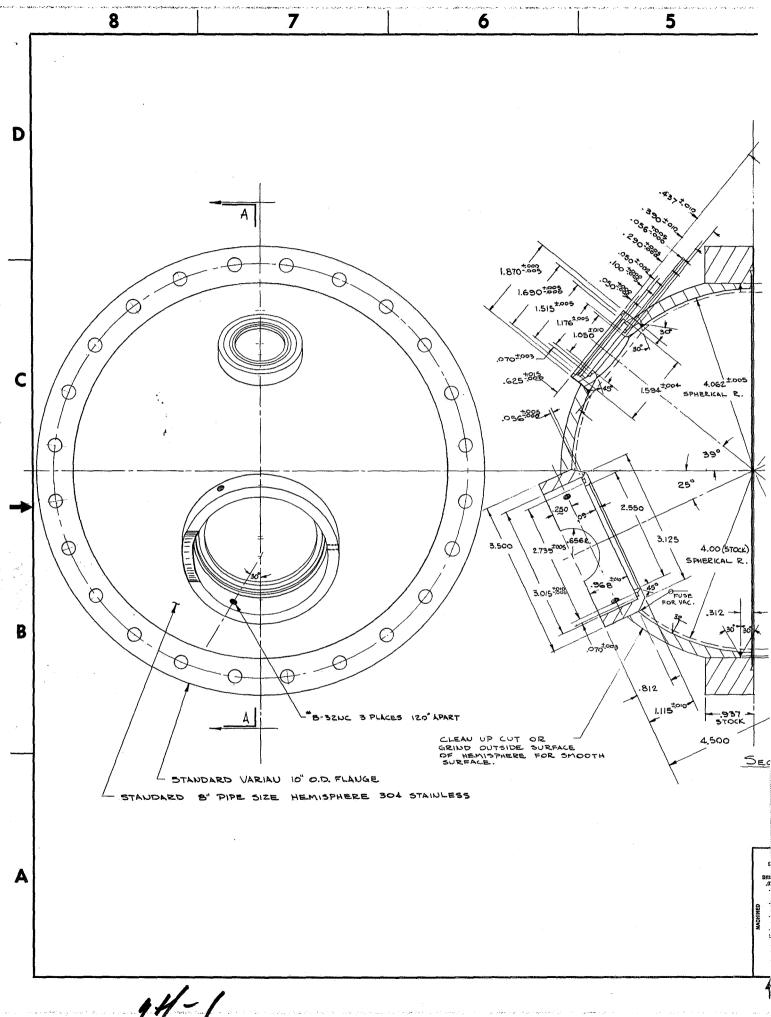
DO NOT SCALE DRAWING

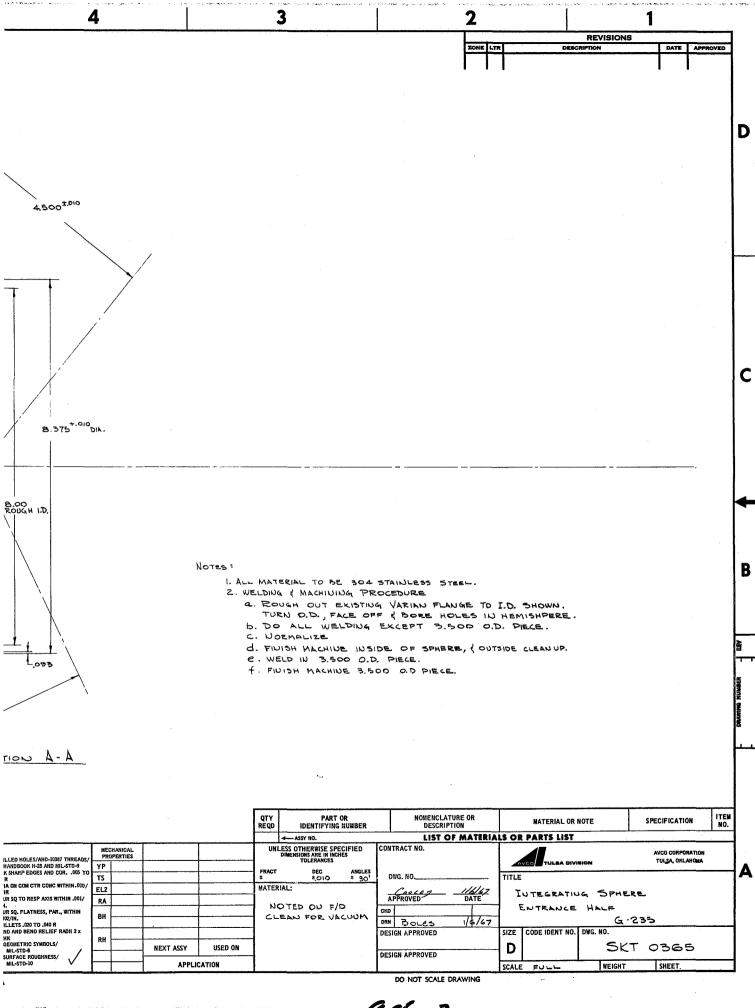


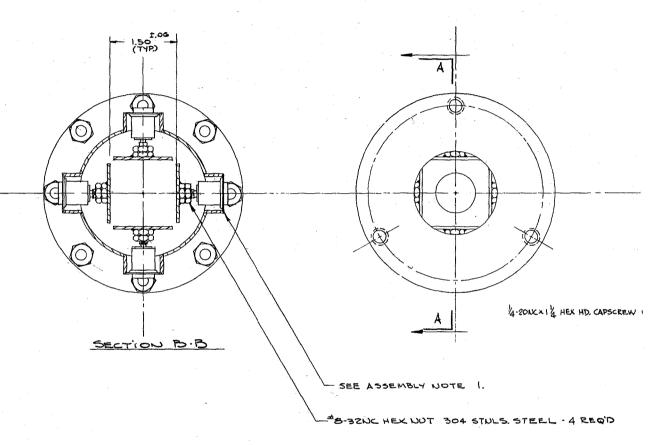












NOTES

D

C

B

A

- Notemble the Locking But (8-32NC) outo the Insulator.

 WITH THE POLE PIECE- (5KT 0374) INSERTED INTO THE HOUSING (5KT 0371),

 SCREW THE INSULATOR INTO THE NUT WELDED ONTO THE POLE PIECE

 PLATE. DO NOT BOTTOM THE THREADS. LEAVE 2 THREADS OFF

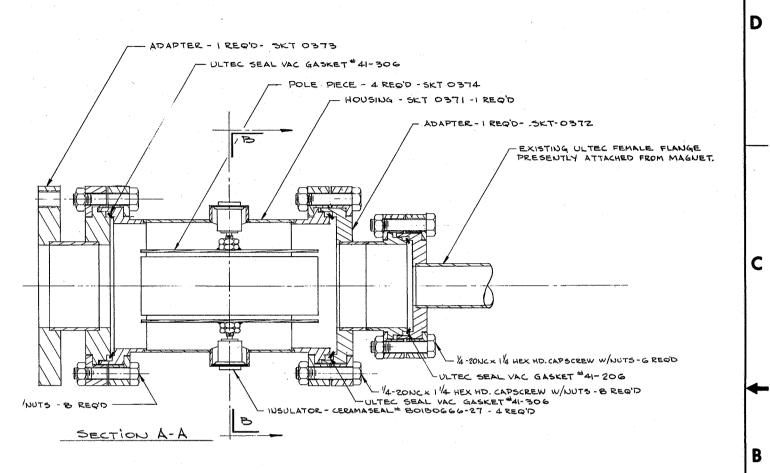
 BOTTOM, OR WHATEVER IS NECESSARY TO MAKE THE 1.50"DIMENSION.

 SILVER SOLDER THE INSULATOR ONTO THE HOUSING.

 TIGHTEN 8-32 LOCKING NUT AFTER MAKING THE POLE PIECES

 SQUARE & TRUE WITH EACH OTHER.
- 2. ALL HARDWARE TO BE 304 STNLS. STEEL.

		REVISIONS		
-	ZONE LTR	DESCRIPTION	DATE	APPROVED



					QTY REQD	tD	PART (ENTIFYING			NOMENCLA DESCRI			MATERIAL OR	NOTE	SPECIFICATION	NO.
					← ASSY NO. LIST OF MATERIALS OR PARTS LIST											
LED HOLES/AND-10387 THREADS/ ANDBOOK H-28 AND MIL-STD-9	MECHANICAL PROPERTIES		UNLESS OTHERWISE SPECIFIED DIMENSIONS ARE IN INCHES TOLERANCES			CONTRACT NO.			AVCO CORPORATION TULSA, OKLAHOMA							
SHARP EDGES AND COR005 TO					FRACT		DEC ±	ANGLES ±	DW	i. NO		TITLE				
	EL2	2 MATERIAL:						Cooley 1/18/67				QUADRAPOLE ASSEMBLY				
SQ TO RESP AXIS WITHIN .001/	IM .001/ RA							APPROVED DATE			10-SITU REFLECTOMETER SYSTEM					
SQ. FLATNESS, PAR., WITHIN	Вн				1				CKD			1	0	(-235		
ETS .020 TO .040 R									DRN	BOLES	1/17/67				G 233	
AND BEND RELIEF RADII 2 x									DESIG	APPROVED		SIZE	CODE IDENT NO.	DWG. NO.		
OMETRIC SYMBOLS/	RH		NEXT ASSY	USED ON	1							D		5	KT 0370)
RFACE ROUGHNESS/			Appl II	CATION	1				DESIG	N APPROVED		SCALI	FULL	WEIGHT	SHEET	

